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Objectives in SOW:

- 1 Prepare replication samples of electroless nickel coated aluminum. Determine process requirements for plating XRCF test optic.
- 2) Prepare and assemble plating equipment required to process a demonstration optic.
- 3) Characterize mandrels, replicas and test samples for residual stress, surface contamination and surface roughness and figure using equipment at MSFC.
- 4) Provide technical expertise in establishing the processes, procedures, supplies and equipment needed to process the XRCF test optic.
- 5) Reports - Three Quarterly and Final.

1.0 SCOPE AND PURPOSE

Advanced optical systems for applications such as grazing incidence Wolter I x-ray mirror assemblies require extraordinary mirror surfaces in terms of fine surface finish and figure. The impeccable mirror surface is on the inside of the rotational mirror form. One practical method of producing devices with these requirements is to first fabricate an exterior surface for the optical device then replicate that surface to have the inverse component with lightweight characteristics. The replicate optic is not better than the master or mandrel from which it is made. This task is a continuance of previous studies to identify methods and materials for forming these extremely low roughness optical components.

2.0 MANDREL IMPROVEMENT STUDIES

2.1 Selected Procedures for Mandrel Preparation

The actual requirements for the x-ray mirror components are surfaces after replication to be achieved with sub-nanometer surface finish. Ideally the surface should be as smooth as physically possible. This requires careful mandrel preparation and cleaning at the gold deposition step after the mandrel has been fabricated. The electrolytic gold deposition process has been dropped in favor of electron beam melt vacuum deposition gold.

Steps for the production of improved mandrels have been studied on two inch diameter coupons and on the XRCF mandrels which have permitted fabrication of x-ray testable mirrors.

2.1.1 Aluminum Metal Selection and Preparation

Two issues are considered in the selection and preparation of the aluminum mandrel. First the material must be free of residual stress which would cause deformation due to relaxation after machining and also free of inclusions or voids.

It has been determined that aluminum alloy 6061 in T-6 heat treatment condition which is relatively pure aluminum will respond well for mandrel fabrication if the material is a good grade. This material is heat treated a second time after rough machining to eliminate surface stress. This alloy is precipitation hardenable meaning that certain elements are present which are less soluble at lower temperature and exceed equilibrium concentrations upon rapid cooling from the melt. In the 6061 this is primarily the copper and silicon which are quenched into a supersaturated solid solution with both solute and vacancies present. Next the aluminum is heated from about 250 to 450° F depending on material and sample thickness. For the 6061 to be treated to maximize the strength and hardness a temperature of 375° F is typically used to form the maximum pinning sites and highest strength, both yield and ultimate. The time may vary from four to sixteen hours depending on the thickness of the sample. This condition is carefully controlled by the manufacturer. After rough machining prior to diamond machining the material is taken to 375° F for one hour to relieve stress due to severe deformation at the surface. For large pieces requiring maximum stability, two or more cycles of LN₂ cooling and subsequent immersion in hot water may be specified. For this operation the preferred method is to suspend the part above the liquid nitrogen for ten minutes or until the part is uniformly precooled. After ten minutes the part is immersed until the nitrogen ceases boiling. At this time it is removed and immersed in boiling hot water until the part is at the boiling temperature. This process may be repeated for large or thick pieces. This step will reverse the stored stress from the original heat treatment quenching cycle from the solution heat treatment. Ideally the aluminum is overaged slightly prior to this rather severe process, but not to the extent that the strength and hardness are seriously compromised. An exception may be if the material was severely bent or deformed such as in a forging or hot forming operation. In this case there may be no alternative other than to reduce the mechanical properties by overaging to eliminate the residual stress.

2.1.2 Material Defects

The mandrel plated coating may possibly contain defects from at least three major categories:

- 1) Manifested by the original aluminum material
- 2) Induced by the plating process or other chemical operations
- 3) Induced by the polishing operations.

These defects may ultimately be in the form of loosely included material which will be lifted by the gold after replication.

Known examples of uncontrolled defect sites include porosity or particulates in the base aluminum metal prior to coating, porosity in the nickel due to excessive trapped hydrogen emanating from the metal surface during plating and imbedded or trapped lapping particles, especially diamond, resulting from the polishing.

A difficult issue is the fact that nearly all U.S. aluminum currently produced contains a certain percentile of scrap aluminum. This in turn imparts particulates of contaminating elements such as iron and glass which are within the testing requirements for the alloy. These particles are not solid solution material and can manifest as defects in the diamond turned part and even chip the diamond tool causing chatter marks.

In order for the plating to properly cover these defects it is required that the surface be carefully prepared using a displacement reaction with zinc and the aluminum surface. An additional precaution is to apply a strike coating at low plating current (Faradaic) efficiency to help bridge these particles. We have determined that this is better dealt with by procuring certified aluminum from an established source although a certain amount of impurity particulates may still be present.

Hydrogen can be driven into the lattice structure of the aluminum (or other metals) by etching or stripping a prior coating for rework or improperly etching the aluminum such as chemical milling to change the dimensions. Proper chemical milling using an anodic process is acceptable. For the reaction $H_2 = 2H^+ + 2e^-$ the pressure of the hydrogen is related to the surface potential of the metal and the pH of the solution by $E_0 = 0.000 - 0.0591 \text{ pH} - 0.0295 \log(P_{H_2})$ where E_0 is the surface potential and P_{H_2} is the hydrogen pressure in atmospheres. This can lead to enormous surface pressures of thousands of atmospheres in strongly acid solutions. When a part must be stripped in acid for rework it should also be baked to remove the hydrogen by diffusion prior to replating.

2.1.3 Nickel - Phosphorous Plating Process Selection

Approximately 40 aluminum samples have been single point diamond machined and used for this study. The samples were plated with electroless nickel ranging from 8% to 13% phosphorous by weight. The electroless nickel coatings were either directly polished or re-turned with the single point diamond turning process and then polished. Electroless nickel plated aluminum

samples plated with nominally 11 Wt% phosphorous for surface finish and passivation studies have been completed. The aluminum alloy used is 6061-T6 hardness which is easily machined by single point diamond turning. A portion of the samples were plated on 2021 - T6. Additionally both coupons and selected mandrels were sent to outside vendors by MSFC. This activity will be reported separately by MSFC. Ten samples sent to OCA were plated by their process and seven were polished at OCA with resulting measurements ranging from about 3 to 10 Å rms

A major drawback previously experienced by MSFC was the electroless nickel plating process instability. This has been mitigated by the switch to a different product. The electroless nickel solution selected is the Enthone Corporation ADP-300 (QA) process which to date appears to be stable and consistently produces a given alloy at an acceptable plating rate of about 6 microns per hour. This process has been studied in detail to determine the optimum conditions to achieve phosphorous concentration from about 9% P to about 13% P maximum. Under the proper plating conditions for metal and hypophosphite content, the alloy composition is almost completely dependent on pH. The Enthone plating process has been in service since February 1995 with no major difficulties noted.

Mandrels have been prepared with an alloy range of about 9% to 13% by weight phosphorous. The alloy is controlled by concurrently plating reference strips along with the mandrel. The strips are removed at appropriate times and analyzed to within 0.2% phosphorous by x-ray fluorescence. This yields a value for the instantaneous plating alloy and also the thickness determines the rate up to the time of removal. Appendix I shows typical results accurate to about 0.2% by weight.

This process will produce lapped parts which meet or exceed the required ≤ 10 angstrom rms level when either diamond turned a second time prior to lapping or when directly lapped after plating using either diamond paste or aluminum oxide compounds. It has been shown that the diamond lapping material may leave imbedded diamonds in the substrate. The objectives have now been moved upward to seek a continuous surface of ≤ 5 angstroms (0.5 nanometer) rms. Additional tests have shown that the deposit is readily lapped to sub-nanometer values both on the coupons and on the actual XRCF mandrels when properly diamond turned.

After the electroless nickel plating the mandrel is subjected to a mild heat treatment of 275° F for four hours to improve adhesion of the coating to the aluminum prior to diamond machining of the nickel phosphorous. If a very heavy coating of Ni-P is used, over 0.007 inches in thickness, then it is wise to reduce the temperature to 250° F for a longer period of 16 hours to avoid undue stress in the coating due to CTE mismatch. For this thicker nickel coating the material can be considered to behave as if the material has no plastic response due to the lack of ductility in the "glassy" or nearly amorphous 11% by weight phosphorous alloy which does not yield as readily as the thinner coatings. A stress of about 7000 psi occurs at 275° F in the 10 inch diameter part with a coating in excess of 0.007 inches. If the residual plated stress in the coating adds additional tensile stress it may actually crack or peel off during an adhesion heat treatment of 275° F.

2.1.4 Cleaning Procedures

Foreign material is difficult to remove from an optical surface, particularly from the gold layer within the replicate if the contaminant is from the mandrel. This difficulty is preeminent whether the gold is vacuum evaporated or electroplated onto the mandrel. It is imperative that the polished nickel surface is free of imbedded material and properly cleaned prior to deposition of the gold to avoid lifting any contaminants from the mandrel after replication. The problem of imbedded material on the surface is particularly difficult since lapping with very small particles such as a colloidal solution of silica or graphite may fill defect sites giving the appearance of a better surface. This may lead to removal of the filling material after replication with the gold and the subsequent nickel shell. For studying the electroless nickel polished surface, the use of ESCA, EDX and ion milling have been used to determine the level of contaminants including polishing materials and possible plating defects (See report by Tom Kante, UAH - 1997).

Different categories of solvents used for removal of lapping compounds from optics were also investigated. OPTICLEAR (TM), a series of nonaqueous solvents, and a hot citric acid based aqueous cleaner, Bowden AW-100 (TM), were evaluated as improved cleaners. Acetone and alcohols absorb atmospheric moisture and impurities leaving residue unless absolute grades of microcircuit quality must be used in a dry and clean environment to minimize contamination. The accepted procedure for cleaning the polished or diamond turned mandrel surfaces includes ultrasonic cleaning in the Bowden citric acid based solution followed by thorough rinsing in de-ionized water and drying with high purity nitrogen. Prior to the deposition of gold the samples were cleaned by a dilute (2 to 3% weight) rinse with ammonium hydroxide, high purity water rinse followed by blowing dry with high purity nitrogen. This was followed by drag wiping with electronic grade acetone on lens tissues followed by blowing dry with high purity nitrogen a second time.

2.2 Deposited Gold for Replicate Optical Surface

Two methods of depositing the gold reflective surface for the mirror have been investigated. The first is to deposit gold using an electron beam evaporation process in a vacuum system. The second is to passivate the nickel surface and deposit gold by electrodeposition from an aqueous electrolyte. Only the electrolytic gold plating process requires the additional passivation step. It is necessary for the lapped nickel phosphorous surface to be subjected to an anodic electrolytic process to form an oxide containing nickel, phosphorous and oxygen. This is to control the adhesion such that the gold will release readily from the polished mandrel. This process is diffusion controlled such that the nickel from the surface migrates through the forming oxide as the current is applied. As the surface oxide forms and the thickness increases, the potential at the oxide surface increases limiting the thickness of the overall oxide.

The complete process for the electrochemical passivation process is outlined in D.O. 111, May 1995 Final Report.

2.2.1 Electrolytic Gold Deposition Process for Electroless Nickel

A comparison was made by replication of samples from the polished mandrel coating of nickel phosphorous directly using vacuum deposited gold or from the electrochemically oxidized nickel phosphorous, using electrodeposited gold. The samples were replicated on polished electroless nickel substrates and backed with at least 0.5 millimeters of low stress nickel. A fixture was built to hold the circular disk parts during the electroplating to achieve a uniform deposit and uniformly low stress. Pre-cleaning and preparation of the mandrels was accomplished by first a hot acid citrate cleaning, step followed by cathodically reducing any spontaneous oxide for two minutes. The passivation process was accomplished in a Rochelle salt solution using computer control and appeared to be very reliable in that all the shells produced released readily with one exception. This piece had a poor passivation due to the use of the wrong selection of sealing material. In this case the computer process indicated a difference from previous parts but at the time it was not understood. The seal decomposed slightly contaminating the surface and affected the uniformity of the oxidation film causing a portion of the subsequent shell to adhere.

2.2.2 Electron Beam Melted Gold Vacuum Deposition

Gold from the vacuum process was applied directly to the polished mandrels after solvent cleaning and wiping with a dilute solution of ammonium hydroxide. Samples which were cleaned ultrasonically in the citrate cleaner showed definite removal of residual materials from polishing. The samples and mandrels which were subjected to the solvent wiping cleaning and handling processes were potentially not as clean. Omission of the cathodic oxide reduction and the hot citrate cleaning was believed to leave the parts with more contamination which could be transferred to the gold later. This subject must still be addressed in the next task.

Results of these tests showed that the evaporated gold from EB-53 produced high quality replicates as did the electrolytic gold, although due to vacuum chamber downtime the number of tests was limited to save time. Samples plated in an alternate vacuum chamber were not superior to the electrolytic deposited gold with about 10 - 12 angstroms typical for each although the original surface of the mandrel had been measured at 6 - 9 angstroms rms. The better samples from the EB-53 vacuum deposited on the better substrates were less than 10 angstroms rms at the gold surface after plating the nickel. All samples measured were from monitored mandrel surfaces. The conclusions to date indicate that if a chamber of sufficient size is available and if adequate cleanliness is observed then the vacuum gold is preferred over the electrolytic process due to convenience and purity of deposit achieved. Also with only one unexplained exception the vacuum deposited gold has released from the electroless nickel easily in subsequent processing of the shell.

2.3 Advanced Materials for Mandrels

Additional tests were conducted on a variety of materials to determine the suitability of refractory coatings of conductive ceramics for applications of replicated optics. Earlier tests on float glass and a silicon carbide reference piece have shown that subnanometer surfaces can be accurately replicated.

2.3.1 RF Sputtered Conductive Oxides, Carbides and Refractory Metals for Improved Mandrels

A mandrel coated with a very hard amorphous or microcrystalline ceramic material which conducts in an electrolyte, may provide a more desirable substrate for the fabrication of replicated x-ray quality mirrors than electroless nickel as the interface to the gold, if it can be deposited or polished to the same or better quality. This has been demonstrated with silicon carbide which has been polished to a surface finish of about one angstrom rms by noted researchers. If such a substrate were non-conductive the first surface which replicates the mandrel could still be applied by vacuum coating procedures so long as proper separation were possible and the current for nickel plating could be properly applied without damaging the coating. Also if the entire mandrel were made of a material which was lower thermal expansion than the electroformed nickel shell then the release could be achieved by heating rather than cooling the system thus eliminating the condensation issues.

Indium tin oxide, chromium carbide, silicon carbide and tungsten carbide are candidate conductive hard ceramic coatings which should be possible to achieve at lower substrate temperature using RF sputtering or electron beam melting procedures for vacuum deposition.

All four coatings will survive the chemical environment for plating electrolytic gold and should be adherent to the polished electroless nickel. Molybdenum is the selected hard metal deposit to be coated over polished electroless nickel/aluminum and glass slide substrates. An attempt to plate a high molybdenum alloy containing trace titanium and zirconium (TZM) was made using E-beam heating of the target but the process used produced a very poor coating. It is felt that a much better molybdenum coating is achievable for the replication study. Samples of pure molybdenum on glass have been coated by Dr. M. Wilson of UAH and were very smooth and coherent with surfaces of float glass at a few angstroms rms being essentially preserved on the back surface of the Molybdenum. These samples were not available to this project.

Samples of indium tin oxide coated glass were obtained from Dr. Palmer Peters and measured by WYKO interferometry. The coatings were also measured for conductivity and ranged from a high of 370 to 400 ohms/square to a low of 60 to 70 ohms/square and the roughness increased accordingly with the lower resistance a function of the thicker deposit. The smoother coatings were about 8 - 15 angstroms on the average. The electroplated nickel was directly applied to the ITO and separated with surfaces very nearly matching the starting values. Attempts to polish the ITO with 0.1 micron alumina resulted in rapid removal of the coating. It was not possible to electroplate gold directly on the ITO samples due to the high cathodic overpotential of the ITO compared to the gold process used.

Samples of vacuum deposited silicon carbide on glass were also obtained from Dr. Peters. These samples measured about 7 - 10 angstroms rms by WYKO and did not conduct sufficiently well to be electroplated with nickel or gold. The use of a vacuum deposited gold will be required if this type coating is used.

A sample of polished silicon carbide furnished by Morton International was used as a measure-

ment standard for the UAH and MSFC WYKO instruments and also the MSFC AFM. This sample appears to consistently return between 0.9 and 3.2 angstroms rms depending on the measurement method and location on the sample.

A plating replicate sample was made from this piece and it was found to be sufficiently conductive to support the nickel plating direct but again not the gold. The sample was coated by evaporation with gold and electroplated with nickel. The deposit was readily separated by using increased temperature differential but without any prior passivation process. The replicated gold/nickel surface was about 5 angstroms rms with some of the disturbance apparently distortion. Measurements of the silicon carbide surface after the replication revealed no change.

2.3.2 Physical Vapor Deposited Conductive Nitrides and Carbides

Additional very desirable coatings may follow from the tool coating industry established by Balzers Inc. The capability to coat tools with titanium and chromium nitrides and carbides is well established and a very large commercial business. These coatings are very hard, electrically conductive and inert chemically. The major drawback is that by the presently used physical vapor deposition (PVD) process, the coatings must be deposited at about 950 degrees fahrenheit. Preliminary tests on electroless nickel plated aluminum coupons were not successful due to the higher expansion of the aluminum which was encapsulated by the nickel. When the samples were heated to the required temperature the nickel cracked and flaked off. Subsequent substrates were 410 stainless steel pieces polished to about 10-12 angstroms rms.

The Balzers engineer provided at no cost, samples of four coatings on steel for evaluation. The samples are TiN (titanium nitride), TiCN (titanium carbon {or carbo} nitride), Cr_3C_2 (chromium carbide) and CrN (chromium nitride). The substrates were not prepared optically and as such the coatings were accordingly very rough as optics. The deposits are very thick at about 3 - 5 micrometers, and indeed are extremely durable. A sample of the Balzers titanium nitride coating on steel was selected for polishing. After about one hour of polishing with 3 micrometer alumina it was apparent that the sample was too rough to achieve a respectable surface by hand polishing in that fashion. Subsequent tests showed that silicon carbide sandpaper up to 400 grit would eventually move the surface towards an improved quality. Diamond or alumina in excess of 9 microns, used as loose abrasive, caused chipping and brittle failure of the titanium nitride coatings. However after about two to three hours of hand polishing with successively finer grit SiC paper down to 2000 mesh, the sample was hand polished with 3 micron alumina followed by 1, 0.3 and 0.1 micron alumina for about 30 minutes each. The surface still was not penetrated to the steel which is considered phenomenal.

WYKO measurements showed this crude polishing process on a very rough starting sample had produced between 5 and 12 angstroms rms surface finish. There was evidence of shallow pitting in the sample surface, possibly due to the substrate. This sample has also been used for a replication test and the material was found to be sufficiently conductive to support both nickel and gold deposition directly. Samples of 410 stainless steel have been prepared by conventional machining followed by heat treating in three stages from 1000 to 1850 degrees fahrenheit and quenching with argon gas.

These parts were then lapped on an automated polisher using successively smaller diamond abrasives to rapidly produce surfaces in the regime of ten angstroms rms with many readings achieved near seven or eight angstroms rms. Subsequently 15 samples have been coated (three each) with titanium nitride and carbo-nitride and chromium nitride and carbide. The TiN was coated onto the 410 stainless pieces with two processes. These samples were coated by Balzers in Tonawanda, NY and returned. The TiN samples coated by the Balzers designation "mirror block" process are very reflective and smooth to about 14 angstroms as received. Data on the polishing and measurements was performed on UAH D.O. 139 monitored by Dr. Michele Wilson of UAH/CAO. These samples were used to produce replicates of gold/nickel surfaces similar to the electroless nickel.

Two samples of TiN coated stainless steel from this group of parts were electroplated with nickel about ten times each. Also each was on one occasion plated with gold/nickel. The samples were electroplated with gold about one micron thick followed by a minimum 50 microns of nickel which was then readily removable. No special activation processes were applied other than to clean the sample prior to plating. The resulting replicates were about the same as the starting inspection of the substrate at about 12 angstroms rms. To date no samples better than about 12 angstroms rms have been produced on the tool coating of TiN from the PVD process. This was true for both the thicker deposits which were lapped and the thinner "mirror block" samples. In all cases the replicate returned was nearly identical to the starting material with no evidence of degradation of the ceramic surface.

A series of smaller samples have been prepared for additional vendors and for polishing tests. The intention is to develop similar coatings by using a combination of electron beam evaporation and ionization of nitrogen at lower temperature to coat nickel plated aluminum with titanium nitride. This would permit the refractory conductive coatings to be applied to lower temperature sensitivity substrates such as the diamond turned aluminum and electroless nickel coated aluminum pieces which could much more readily be fabricated in the shapes and accuracy required for the x-ray mirror mandrels. At this time however, support for this activity is not available.

2.3.3 Alternate Electrodeposited Coatings for Mandrels

Electroless nickel 11% phosphorous alloy, which is nearly amorphous, is presently used to coat aluminum for the purpose of producing mirror surfaces. A third approach to fabricating improved mandrels is to develop an electrodeposited coating which will support the sub-nanometer surface requirements. A deposit is desired which can be electrodeposited over aluminum or other materials and single point diamond machined and polished. The electrolytic processes are typically far less troublesome than the highly volatile, hot electroless solutions which spontaneously decomposes with use. Note that this is an addition to the original task not in the SOW so the effort is non-interfering in scope.

The electroless nickel plating processes are not as easily applied as an electrolytic coating. This is due to a number of reasons but particularly true due to the need to replace the nickel and phosphorous chemically rather than from a soluble anode system. Also the deposit is attained at a

very high temperature, near boiling, which requires special equipment and handling. The solution constantly decomposes such that after a fixed amount of deposit is acquired per unit volume of solution then the solution must be salvaged for nickel or sent to a chemical waste treatment facility. Unfortunately, the electrolytic pure nickel plating processes do not produce a deposit which is readily diamond turned and polished. Crystalline deposits of high purity nickel form carbides with the diamond tool cutting edge and very rapidly degrades the edge surface of the tool. If a nearly amorphous electrolytic nickel deposit can be attained with the use of appropriate additives it should be possible to diamond machine the material without significant damage to the tools in a normal tool life span. Previous attempts by several researchers to electrodeposit nickel phosphorous have not been successful in producing high quality deposits, however recently Tech-Metals of Columbus, OH announced that they can produce such a deposit. NASA MSFC is pursuing a study of this material outside this contract to determine the suitability for fabrication of large mandrels and mandrels requiring thicker deposits. It has been possible to achieve a very fine grain structure nickel through the use of additives other than phosphorous. This type deposit was explored briefly for compatibility with optical fabrication processes.

A solution of nickel sulfamate containing two organic additives, 1,3,6 naphthalene trisulfonic acid and coumarin was used to plate a small cylinder. This deposit was then turned by UAH using a conventional zero rake diamond tool. The surface was not as good as turned nickel phosphorous and returned only about 300 angstroms surface finish, however the tool was not damaged as is the case with pure nickel. Polishing of this surface did improve the surface rapidly with about 20 - 30 angstroms achieved. No additional time was put into this effort pending results of the commercial source for electrodeposited nickel phosphorous.

2.4 Hybrid Composite / Plating Combinations

Limited efforts on the fabrication of a test mirror using epoxy graphite filament wound components have demonstrated a limited feasibility. Two shells were produced with deposited nickel shells of only 0.15 to 2 millimeters thickness for the application of composite outer shells of filament wound graphite epoxy. These thin nickel electroformed shells were successfully removed from the mandrel without damage. Extended efforts by the European Space Agency on XMM for several years have not yet knowingly resolved all the issues of the extremely tight tolerance requirements with the use of graphite and polymeric materials. The moisture desorption and uptake is readily calculated to provide extended time deformation calculations of at least bars and plates for these materials. The approach taken by ESA has been to fabricate and maintain the components in a dry environment. Due to the extensive effort required to achieve this environmental control and improve on previous work by others, this effort has been halted at MSFC for now.

A second approach considered was to add a substantial amount of graphite fibers to the plated part during the plating process. The procedure produces what is called an electrocomposite. A sample of nickel plated graphite tow was ordered and received. This material was used to determine if an electrocomposite could be formed by plating the graphite into the nickel shell at low stress. This will produce a composite part with high stiffness allowing for much reduced

weight. Two samples on a cylindrical mandrel were produced with the density at about 5.0 grams per cubic centimeter. This is about 56% of the weight of conventional nickel. This type two phase material will be more sensitive to deformation due to thermal effects as the matrix is comprised of two materials with very different thermal properties. No further studies were made on this material due to time constraints.

This contract has also supported some work on Next Generation Space Telescope (NGST) which has lighter replicated segments of a very large mirror system under study. Additional funding to UAH has also been provided by ULTIMA for a study of the potential to replicate extremely lightweight components. The goals are such that the composite hybrids are potential candidates for this type of on-axis mirror system. A series of 7.5 inch diameter flat mirrors were made for the NGST experiments with a foam metal aluminum backing to be applied by electrojoining or epoxy. The large CTE mismatch is believed to have caused unacceptable print through of the foam metal contact points with tens of waves distortion localized at these points while the overall flatness appeared to match the mandrel.

3.0 XRCF CALIBRATION X-RAY MIRRORS

The principle effort of this contract has been to fabricate and test small electroformed nickel x-ray mirrors using the procedures derived through the test coupons and prior work. The mirrors were fabricated using in-house MSFC capabilities almost exclusively.

3.1 XRCF Calibration Optic Plating Studies

3.1.1 Hardware

Significant activity has been given to plating tests and hardware to assure that the high quality mandrels fabricated for this effort would not be damaged and that the ultimate quality will be replicated. Nine shells in total were produced from mandrel number 1. The information includes plating parameters and computer control algorithms for assurance. The plating data includes alteration to the anode configuration to achieve better uniformity. This is needed to provide uniform stress under control of the computer. The computer program was updated to allow for shutting off the power supply at a predetermined number of amp-hours of plating. By establishing the plating requirements coulometrically it is possible to predict the average plated thickness of the part. The distribution of the deposit onto the end pieces is such that any heavy plating is not on the actual shell. By placing the anodes close to the part and covering all but a two inch wide slot it has been possible to improve the distribution of the deposit on the central portion of the mandrel to permit the part to be plated within about two or three percentile low or high distribution with respect to the weight averaged normal thickness. This in turn permits accurate control of the distributed stress in the shell of concern without additional shielding. See Appendix II for the presentation of this information.

3.1.2 XRCF Mandrel Development

The weight and thickness measurements also permitted another problem to be realized. The continuity of the end pieces to the central shell mandrel is made by connection through a wave spring. It had been determined earlier that the stainless steel spring would drop significant voltage at the required plating current to lower the deposit mass unacceptably on the central mirror mandrel. Thus the washers were copper and gold overplated to form a better current carrier. However even with the plated washers several plating tests showed lower mass on the central (or mirror) mandrel than predicted or than previous experiments. The cause was oxidation of the aluminum forming a poor connection to the wave springs. Also on at least one occasion the seals leaked allowing nickel plating solution to seep into the interior of the mandrel corroding the connections and lowering the conductivity.

3.1.3 Seals

A study of different seal materials provided better knowledge of seal performance against all of the plating and cleaning materials. Red room temperature vulcanizing (RTV) rubber had been used exclusively until this time. The red material apparently contains iron oxide and permits the material to conduct under certain conditions after plating for extended periods such as the electroforming requires.

Data and samples of several materials were obtained and tested.

Butyl rubber, Urethane (Harkness MP-600), Teflon (TM) and KALREZ (TM) elastomeric Teflon by DuPont and three varieties of RTV were evaluated. The decision was that the Teflon and KALREZ were clearly chemically superior and the KALREZ is most desirable.

The butyl rubber product survived well in the nickel plating exclusive of other processes, but in the alkaline solution for preparation to electroplate gold the seal failed. Electroplated gold was used since the vacuum system was being moved at this time. The Urethane MP-600 behaved well in the nickel but the data sheets did not indicate that this material would suffice in the other processes. Pure Teflon (R) was of course very inert in all the chemical processes of interest but suffered from lack of an acceptable sealing surface. Samples were lapped and appeared to seal against leakage but the edge of the deposit grew over the mandrel in the form of fine whiskers within very small striations along the Teflon seal. This in turn caused difficulty in removal of the shell. A number of seal manufacturers and distributors were contacted and the recommendation for a high value part was to use the newly developed Teflon elastomer from DuPont. This material is called KALREZ and bears a price tag of more than \$25 per square inch with a single piece minimum purchase of 6" X 6" costing almost \$1000.00. This material was tested along with the Harkness Urethane MP-600 in just nickel plating since the vacuum deposited gold eliminates the need for the other processes. Both performed extremely well with the KALREZ actually forming a more permanent bond which rendered it somewhat more difficult to remove later.

As long as the only plating is the electrolytic nickel, the Urethane MP-600 is desirable. Other grades of the Harkness urethane stock material were less acceptable with one of the harder grades

being unacceptable in that the plating climbed over the edge faster than on the RTV samples. For the electroless nickel cleaning, activation and plating processes, the red RTV appears to be superior to all but the KALREZ. The choice is to use the RTV for the electroless processes and the Harkness urethane for the electrolytic nickel plating. Teflon is still used for the vacuum coating process to separate the end caps from the central mandrel since the gold can be removed with diluted aqua regia and the seals reused any number of times. This step requires that the mandrel be disassembled and reassembled with the urethane seals with the delicate gold coating applied.

3.1.4 Nickel Plating

Nickel plating thickness uniformity and zero stress conditions were studied using a part which was rough machined. This required measurements of the current density with the QMI current probe used on AXAF-S and fixture or anode adjustments to assure the uniformity of the current density. A plating fixture was purchased to plate the XRCF part horizontally in the electrolytic plating process. This is to insure that the edge effects are minimized to eliminate the need to trim the ends of the mirror by machining or grinding as before. The plating proceeded with very good uniformity with less than 10 percentile deviation. See Appendix III.

A second and third electroless nickel plated mandrel were diamond turned using the new Pressotech turning center and achieved 25 angstroms rms directly off the machine. These pieces were polished to a surface of 3 - 6 angstroms rms using only alumina and to date are the best x-ray mirror mandrels produced by MSFC. These mandrels were not plated until all data from the previous testing had been compiled and the optimum conditions were determined.

Two preferred optical components have been completed and tested. The first was made on Mandrel 03 with gold plated by EB lab personnel and the second was the same mandrel with the gold coated by R. DeHaye. The results of all the x-ray test data will be compiled and reported by those responsible under separate cover. The preliminary results indicate that indeed both Shell 01 and 02 of Mandrel 03 are extremely good quality with no discernable differences observed. This demonstrates a repeatable process.

3.2 Separation of Electroformed Nickel Shells

The first test mandrel was coated by evaporated gold and plated with one millimeter nickel. This part was then used to test the new separation fixture. The separation of the electroformed shell proceeded well using the new extraction fixture designed and built by MSFC. A small dimple in the part due to a machining error caused a slight problem in removing the shell and the temperature had to be reduced below that of the actual release. The removal was performed in a dry glove box and the moisture condensed was minimal although some frost occurred due to the extremely low temperature (ca -40 degrees C). The surface appeared to be clean and free of condensed films. Due to computer problems, the stress was not controlled continuously throughout the plating run. A second gold coating was evaporated onto the part and the test repeated. Again the computer halted and failed to retain data. The program was then reviewed and modified to compile the hardware configuration prior to establishing memory commitments.

This appears to have resolved the issue although storm interference may also have had a role in the second program failure. The computer and stress monitor are on a UPS but the plating power supply and pumps are not due to the high current demand.

Subsequent plating tests with mandrel SN 1, involved testing of the automated passivation setup. Three tests were successful in that the passivation control by the computer produced the appropriate surface for the electrodeposition of the gold without excessive or insufficient adhesion. The mandrel was unfortunately damaged during the fifth plating test due to the leaching of elastomers from the butyl rubber seals under test. The alkaline solution used to clean and remove oxides from the mandrel prior to passivation apparently leached the gasket material (See section 2.2.1). This caused poor adherence of the gold in the proximity of the seals which allowed nickel plating solution to leak in between the shell and the mandrel causing damage to the mandrel. A sixth shell was prepared to continue testing of the shell thickness uniformity. The polarization behavior for the passivation clearly identified that the surface was not the same as usual. A seventh plating test on this mandrel was the final test on the mandrel due to the damage and the completion of the next mandrel.

3.3 Weight Reduction Studies

The next phase of our efforts was to reduce the optic weight. Several approaches are considered and action has commenced now that suitable x-ray optics have been fabricated. One method of producing light weight optics is to simply calculate and build the thinnest wall shell which still performs well. The next is to devise a reinforcement method.

Studies include a closed solution calculation of the shell deformation as it rests on a flat surface by UAH and a finite element analysis of the shell resting on two V-block pads by NASA using NASTRAN. The two methods are in good agreement and demonstrate that at 0.75 millimeters thickness the shell is stable to less than one micron sag by self weight under either condition. A geometric increase in the sag is observed as the shell thickness decreases to the lowest value observed at about 0.15 millimeter sag at 0.1 millimeter thickness. The interesting observation is that the top of the part apparently sags below the surrounding material slightly in the thinnest cases. This was apparent in both analyses.

Next a simulation of the stiffening effects of slender rings was performed again by two methods. Comparative results are not yet available but an analysis of the rings with similar loads has been done to show at least stiffness at the ring site. For three rings of about 0.5 millimeter thickness and 1.5 millimeter height the deformation of the shell at the ring is returned to the near zero value observed for the thicker shells. There is still some longitudinal sag which is not supported and this appears to be about equivalent to the circumferential sag in magnitude for a shell of a given thickness. This probably means that for 1-G loading it will be necessary to support the shell at more than two points.

Methods of reinforcement studied also include the addition of a composite shell to a thin wall electroform prior to removal from the mandrel as described previously. This is underway by MSFC personnel. The other is to electroform or EDM the small lightweight rings as designed,

consistent with the design of spiders to hold separate shells in an assembly later and reinforce the thin shells by attaching the ring to the shell prior to removal from the mandrel.

The ring can be metallurgically joined by deposition of the nickel to a machined support. This process is known as electrojoining and has been used to join materials sensitive to welding temperatures and also optical electroformed pieces which could not be brazed or welded without distortion.

The design and fabrication of the lighter weight optics has been started based on the sag calculations performed on NASTRAN by MSFC. The data has been input into actual mirror design programs and is under study. The simpler calculations of the predicted sag in a cylindrical shell have been observed to verify the results. The sag is reduced in a cubic fashion as the shell becomes thicker. At the thickness of 0.75 millimeters used presently, the sag is reduced to about one micron or so.

Three thin wall parts have been fabricated to date. One with about 0.15 millimeter thickness on mandrel 3 and two of 0.1 millimeters thickness. One was made on mandrel 3 and the other on mandrel 1. The separation of one of the thin shells has been accomplished with relatively little difficulty.

A set of three rings was made by electroforming nickel using a side forming method from disk shaped mandrels. The rings were next placed over the thin 0.1 mm shell on mandrel 1 and the part was cleaned and returned to the plating solution. An additional 0.05 mm of nickel was plated to join the rings to the shell. The results appear to be very encouraging. The shell total thickness is 0.15 mm now and the rings appear to have joined very smoothly.

The X-ray testing of the ring supported shells is not yet complete. Data from the thin shells from mandrel 3 are scheduled to be analyzed in July 1997. Additional fixturing and rings for supporting thin shells were studied during the fourth quarter of this contract.

Appendix II, figures 1 and 2 shows the mass and supported shell deflection characteristics for supporting rings of two thicknesses.

Figure 3 shows the deflection of an unsupported shell versus thickness.

Figure 4 shows the increased moment of inertia (stiffness) of the ring plus the shell for a thin ring of 0.010 inch thickness.

Figure 5 shows the exaggerated deflection at increasing amplification for a 0.006 inch (0.015 mm) thick shell simply supported by a flat surface. Note that the deflection appears to be greater at both the top center and bottom center than at points immediately adjacent. This is somewhat similar to the MSFC FEA analysis on two V-blocks.

4.0 ADDITIONAL ELECTROCHEMISTRY STUDIES

In order to achieve the overall goals of producing electroformed x-ray or any other optical components, it is imperative that the deposition process be controlled in every aspect. The weight and distortion of electroformed nickel components is an issue unless the deposit can be produced with very low, ideally zero, internal stress throughout in thin sections. This can only be accomplished if two rules are strictly followed:

- 1) The deposit must be current density controlled above and below the neutral plating rate stress point
- 2) The deposit must be uniformly deposited in thickness to assure the same current density over all of the part.

The stress in the deposit is controlled by minute changes in the acicular structure of the nickel grain boundaries developed during crystallization. Many solutions support the deposition of nickel from the point of view of the anion forming the ionic complex. None are known to produce a free standing, high purity nickel with low stress better than the sulfamate process introduced many years ago. A diffusion process controlling the incorporation of a sub-ppm concentration of nickel sulfide in the grain boundaries is believed by most researchers in the field, to be responsible for the control mechanism. By plating at different rates the concentration of these grain boundary striations can be controlled since the formation of the nickel sulfide is diffusion limited while the deposition rate of the nickel is strictly speaking, mass transport limited by the available current according to Faraday's Law. Therefore the increase of current density will cause a lower ratio of the enlarged grain boundaries to form and will permit a more tensile deposit while the lowering of current density will allow a higher formation opportunity for the enlarged grain boundaries causing a lowering of the stress.

Unfortunately it is not this simple. Many other factors must apply to allow observance of the two cardinal rules above. The solution condition especially pH and temperature also immediately affect the stress control. A drift in other parameters can cause a drift in the stress as well. In particular the rate of change of potential at the nickel surface with the change in current density must have the same value globally on the part. This is particularly difficult since at least three processes are controlling transport phenomena.

In simplified format these are:

- 1) Solution resistance or conductivity
- 2) Rate of exchange of nickel and nickel sulfide through the immediate boundary layer next to the depositing mirror
- 3) Potential field due to spacing or shielding of the immediate location on the mirror with respect to the anode.

While it is certainly not our intent to reinvent the process of nickel plating, it is mandatory that those involved in the process of electroforming mirrors are cognizant of the parametric relationships controlling the deposit quality including perhaps foremost the intrinsic stress.

The static solution conductivity per se, is relatively constant over the considerations required but is related to item 2 in that higher or lower agitation can affect the rate of exchange through the boundary layer consisting actually of two boundaries and usually called for that reason the double layer. The potential field is related to the current density by the rate of change of the potential to the current density as mentioned. The situation in general is that as the end of the part is considered the current density increases geometrically based on physical arrangement of the cathode and anode. For all but the most simple configurations this is very difficult to calculate. Ideally this is constant for a cylindrical part if the ends of the cylinder are exactly coincident with a conforming cylindrical anode and if the ends are completely insulated across to the anode. This does not apply for the real situation at hand since it is all but impossible to achieve this arrangement. Fortunately by inserting insulating shields between the high current density areas on the cathode and the anode it is possible to "shape" the potential field from which the current density is also controlled in accordance to condition 3 above.

4.1 Computer Programming of Electrochemical Behavior

Recently two major computational efforts have emerged as tools to assist in the design of this shielding. One is a comprehensive PC based program from El-Chem in Ohio which has been developed by Dr. Uzeil Landau of Case Western University. This program has been purchased by MSFC for use on this effort. The second was written by Oak Ridge National Laboratories which produces a three dimensional model and considers solution fluid properties such as agitation over the surface considered. This program by Gary Giles and Jon Bullock will not run on a PC and is not available to the public. They have produced and presented a cursory model for comparison to the El-Chem results. A third much less comprehensive program written by D. Engelhaupt of UAH, allows for the design of a perforated shield using electrochemical impedance considerations but requires at least one test piece prior to iteration of the design values for the number of holes, spacing, thickness and diameter of the perforations. Additionally after this program was written, an article was published by NEED NAME HERE using a somewhat simplified approach but requiring instead of the test piece, a computer boundary element evaluation based strictly on conductivity as input. This approach is not believed to have been used in a separate program

4.2 Computer Aided Design of Electrochemical Cells

This commercial program was purchased to allow simulation of the overall plating cell for new designs such as the 10 and 20 inch mandrels under fabrication. The program operated under DOS on a PC with three non-interactive modules when purchased. Since this time a new version plus additional geometry editing support is available and runs under Windows 95 (TM). This software has been used to model the 10 inch mandrel. It was found that significant errors in the results were obtained with the software default values for the electrochemical properties of sulfamate nickel plating.

Oak Ridge National Laboratory Computational Comparison:

A request was made to Oak Ridge National Laboratories to use their more sophisticated three dimensional program to model the 10 inch part and determine preferred shielding. The results were also in error with the parameters available. A brief literature search determined that it would be best to make our own measurements of certain electrochemical parameter values. The log current density slope versus the applied potential and the current value at which a substrate switches from cathodic to anodic were required. Also the value available for the conductivity of the solution was in error.

The measurements were made in the MSFC corrosion laboratory and then used by both UAH and ORNL respectively. Although the ORNL 3-D model is far more sophisticated, the L-Chem models compared very well in 2-D. This effort has allowed the development of what appears to be an appropriate shielding approach once the appropriate input parameters were available. Considerable additional effort must be put into this effort to produce mature 3-D results. The effort by ORNL would need to be supported in some way in order for them to finish a complete three dimensional model. However the work completed to date is very impressive. See Appendix IV for additional electrochemical data and samples of the deposit thickness achieved with different candidate shielding approaches.

5.0 FUTURE WORK RECOMMENDATIONS

Future work must proceed on the process development for the 10 inch and 20 inch mandrels. The computer modeling of the shielding requirements should be completed prior to the gold deposition to avoid delays. Testing of the cleaning process of the electroless nickel prior to gold deposition also remains. Twelve test pieces have been fabricated for this test.

Additional advanced development is required to produce lighter components. This includes a study of both improved material properties and geometric stiffening methods and materials. The use of electrocomposites, deposited alloys and electrojoining procedures should be considered. This additional support will also be applicable to NGST and other replicated optics programs.

This is considered on the stiffening rings, that is to move the moment of inertia of the shell outwards to stiffen the thin shell with the addition of minimum weight. In the case of the NGST and Ultima, not only geometric stiffening such as the use of ribs but increasing the thickness of the mirrors with high stiffness ultra-lightweight cellular material should be considered. In this case the thickness is not so critical as in the grazing incidence x-ray shells and represents an entirely different category of fabrication issues. Since the specific stiffness of most designs is a cubic function of the thickness of a fixed material and linear with density, considerable improvement is possible with cellular construction techniques. By attaching a thick open cell material to a thin replicated mirror it should be possible to achieve less than 5 Kg/M² areal weight with good stability. The issues become more serious with regards to CTE matching and print through of bonded areas at isolated attachment points particularly when large temperature gradients or changes are involved. These procedures have been demonstrated for years and should be explored for quality and suitability to perform some of these tasks.

A review of the following two cases is appropriate:

1) Case for low cross section requirements of the grazing incidence mirrors:

It is necessary to define the maximum thickness of each individual shell in terms of x-ray collection area masking and overall size requirements. The material density and elastic modulus is now critical. For nickel at an elastic modulus of 30 MPsi and a specific gravity of 8.9, the areal weight of a simple shell becomes limited to either the microyield which permits permanent deformation during a fabrication task or during launch or the 1 G self deflection at a given diameter, limiting inspection and assembly, whichever is dominant.

a) Microyield dominant: A change is in order within the material strengthening mechanisms, including the following considerations:

Additional pinning sites

Addition of second or higher phases by alloying or composites

Improved microstructure (eg. smaller grains, acicular structures or lamellae)

The microyield of nickel or alloys with cobalt or iron can be tailored over a factor of at least five. For the electroformed nickel alloys this is on the high end and can be changed at least by a factor of three using known alloying methods. What is not published to my knowledge is extensive methods for controlling the intrinsic stress in such deposits. I have experience in controlling the stress in nickel - iron deposits which have upwards of 200 KSI engineering yield (0.2% offset) and nearly 300 KSI ultimate tensile strengths. This is about 70 to 100 KSI microyield compared to about 30 KSI for conventional electroformed nickel. Similarly the addition of ceramic particles or fibers of sufficiently small diameter can substantially improve the micro yield strength and lower the CTE and density while an increase in the elastic modulus is realized. Either the iron or the composite would allow lighter shells if the microyield is the issue. For the case of the iron I have deposited true invariant expansion (INVAR) material.

b) When the self deflection becomes the design issue:

The bulk thickness must be increased with increased weight

The design must include additional thickness (obscuration) using cellular methods

The material must be changed to a higher specific stiffness selection

A combination such as the composite shell/replicate face may be employed.

The improvement in the specific stiffness of a material depends on alloying or combining with another material. This material usually, not always, has higher elastic modulus and/or lower density. Some exceptions exist such as nickel aluminide which is used to improve the performance of turbine blades but no known deposition process for electroforming this material is known.

Many ceramic cases are known such as aluminum and other oxides, many nitrides and carbides which can be formed into extremely stiff shapes. The replicated surface must be independent in

these cases. A combination of these materials with nickel electroforming is quite possible in the form of the "electrocomposites" or co-deposited ceramics in the nickel. In the case of mirrors the initial thin layer would not be a composite and would be formed from a second process comprising the composite. Although an interest in these materials has been around for about forty years, not much has been done in the actual industrial use of these materials due to lack of understanding and control driving the cost up. Although not applicable as a high stiffness or high strength material, a significant recent development is the better understanding of Teflon - nickel composites used to produce very low coefficient of friction surfaces for sliding components. In this case the adsorption and deposition phenomena are becoming fairly well defined for producing high concentration Teflon deposits of high integrity. Several reports are recently available on the studies. Additional recent attention has been given to the deposition of nickel with aluminum oxide and silicon carbide to improve the mechanical properties beyond the alloys of nickel with other metals. It is required to understand the mechanisms for adsorption of the material into the nickel deposit and for controlling the stress if this is to become useful to us. The inclusion mechanisms are not completely understood at this time, but to be sure the mechanical properties can be improved substantially. It would be of interest to extend the composition methods with the alloy of nickel - iron to achieve several properties simultaneously which might otherwise be mutually exclusive. The use of the composite shells is still viable as a second structure using the thin replicate inner shell.

2) Case for the thickness relatively insensitive (NGST):

a) Improvements in the design to increase the stiffness without additional material (weight)

Cellular material attached to back of replicate
 Material deposition with ultra-lightweight hollow spheres of graphite or ceramic
 Material deposition with lightweight ceramic filler
 Cellular or reinforcement rib construction

b) Combinations of above.

Potential Task:

A task to demonstrate the above within the hours I have to commit might include:

1) Formulation and study of an advanced alloy - composite process

a) Demonstrate a succinct material properties improvement with low risk

Deposit nickel-25% iron alloy with known procedures
 Measure properties and compare to pure nickel

b) Add to the process, a ceramic material to further advance the properties

- c) Demonstrate a two layer replicate plus advanced material deposit wherein the thin lightweight replicate is by more conventional nickel electroform processing.
- 2) Continuation of ongoing NGST / Ultima concepts
 - a) Join materials with cellular structures to the back of the replicate
 - b) Study methods of reducing and eliminating print-through such as forming a partial face prior to joining and filling the cellular structure, then perhaps forming a second back plate prior to removing the filler to achieve the best weight to stiffness ratios.
 - c) Combinations of both advanced materials and attached structures.

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APPENDIX II - XRCF Deflection and Plating Data

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Figure 2 Mass and Deflection of Stiffened Shell - 0.01 inch Ring

Figure 3 Deflection of Shell - Self Weight Deflection Versus Thickness

Figure 4 Stiffness of Support Ring Versus height with 0.006" Shell Loading

Figure 5 Deflection of Shell Resting on Flat Surface

Figure 6 Relative Shell and Cap Weights for Completed Parts

Figure 7 Shear Stress at Shell/Mandrel Interface Due to Cooling

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APPENDIX IV - Electrochemical Study and Current Density Results to Date

Figures 1 and 2 show the vastly different electroforming configurations

Figures 3 through 8 show progressive improvement in distribution of deposit with shielding.

Figures 9 and 10 show the polarization characteristics of the plating solution.

Nickel Plating Parameters

APPENDIX I - Electroless Nickel Data

PERCENTILE PHOSPHOROUS VS pH OF ADP-300

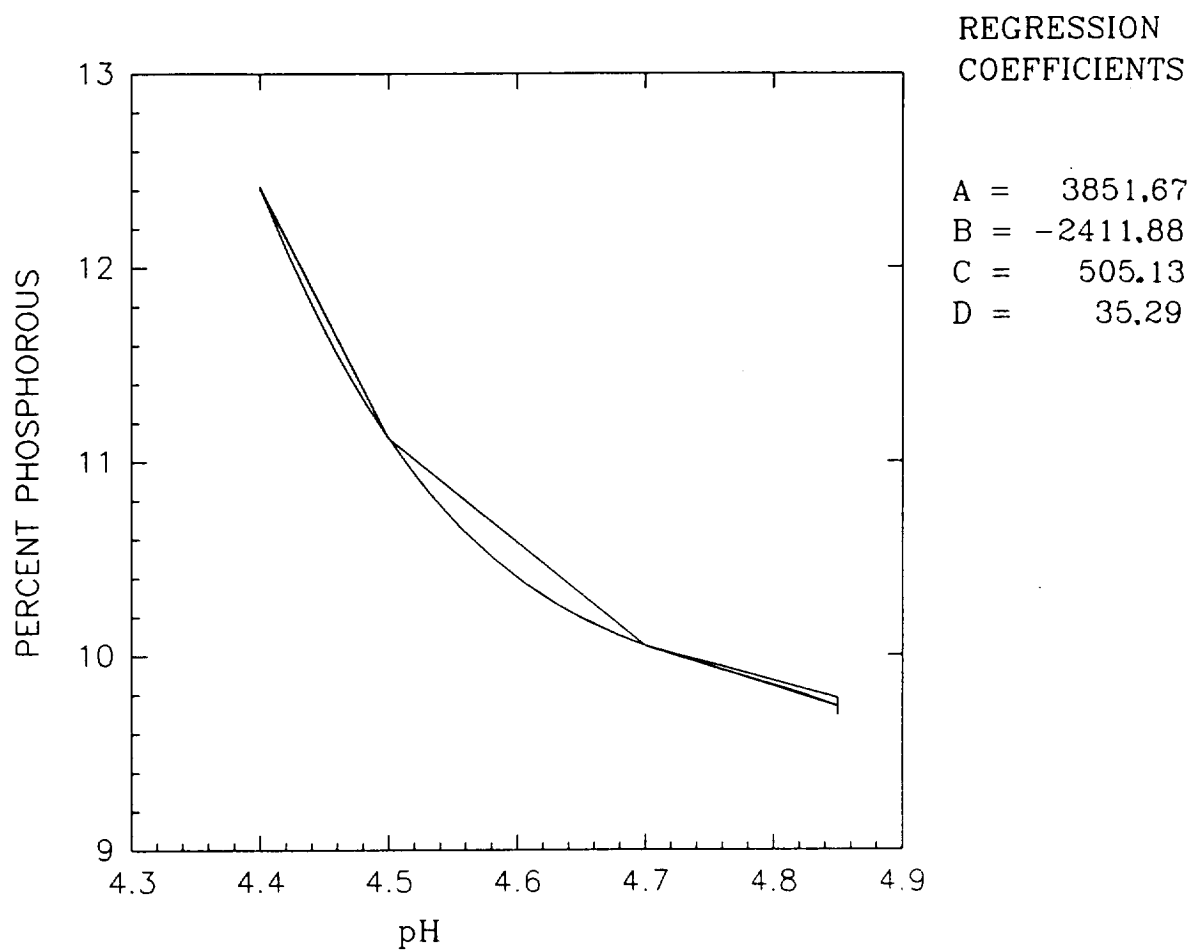


Table 1 - X-ray fluorescence Nickel and Phosphorous Alloy Data

SAMPLE #	Ni(Wt.%)	P(Wt.%)
1	89.10	10.90
2	90.19	9.81
3	88.84	11.16
4	89.05	10.95
5	89.25	10.75
6	89.22	10.78
7	88.34	11.66
8	88.45	11.45
9	89.95	10.05
10	90.40	9.60
11	88.28	11.72
12	89.11	10.89
13	89.12	10.88
14	89.15	10.85
15	88.91	11.09
16	88.28	11.72
17	89.11	10.89
18	89.12	10.88
19	89.15	10.85
20	88.91	11.09
21	88.24	11.76
22	89.29	10.71
23	88.43	11.57
24	88.42	11.58
25	88.77	11.23
26	88.66	11.34
27	88.90	11.10
28	88.65	11.35
29	90.86	9.14
30	90.90	9.10
31	89.64	10.36
32	89.41	10.59
33	89.28	10.72
34	89.34	10.66
35	89.68	10.32
36	89.16	10.84
37	89.08	10.92
38	89.23	10.77

APPENDIX II - XRCF Deflection and Plating Data

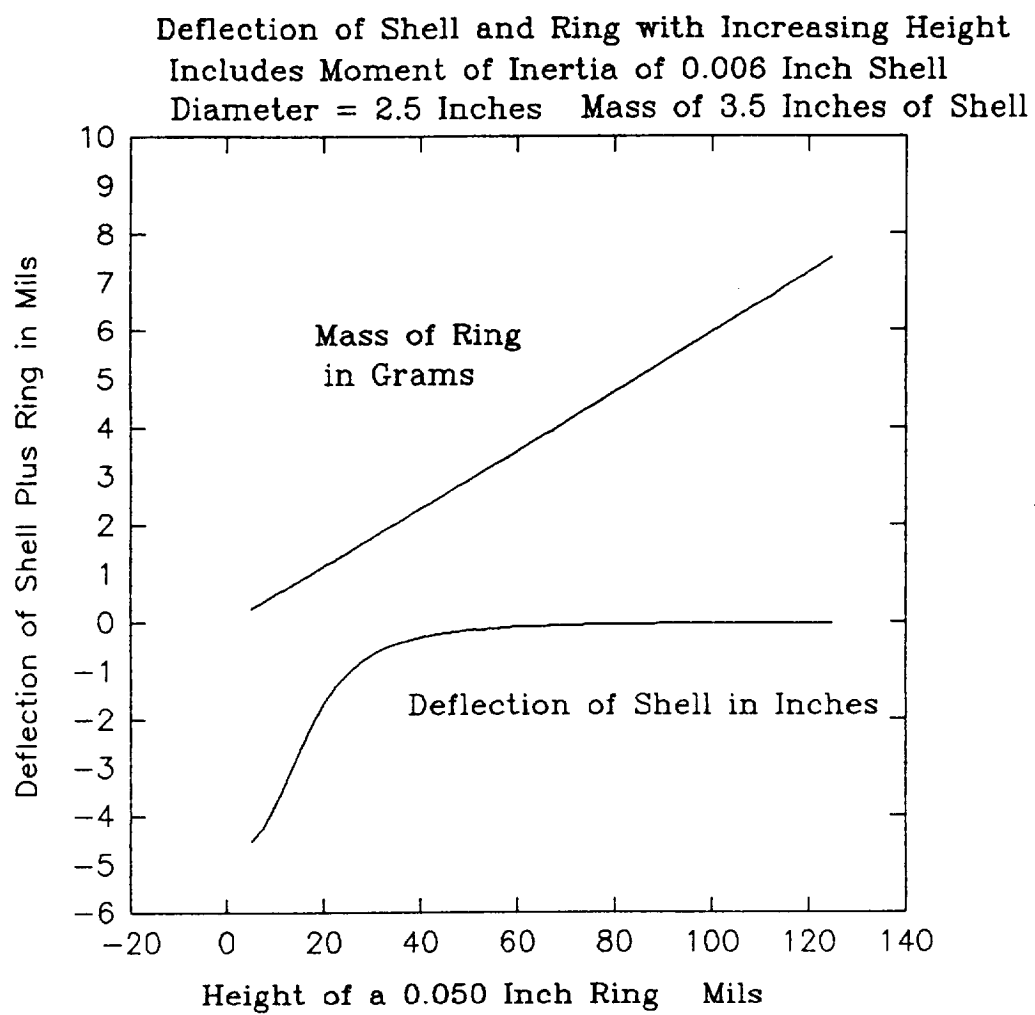


FIGURE 1

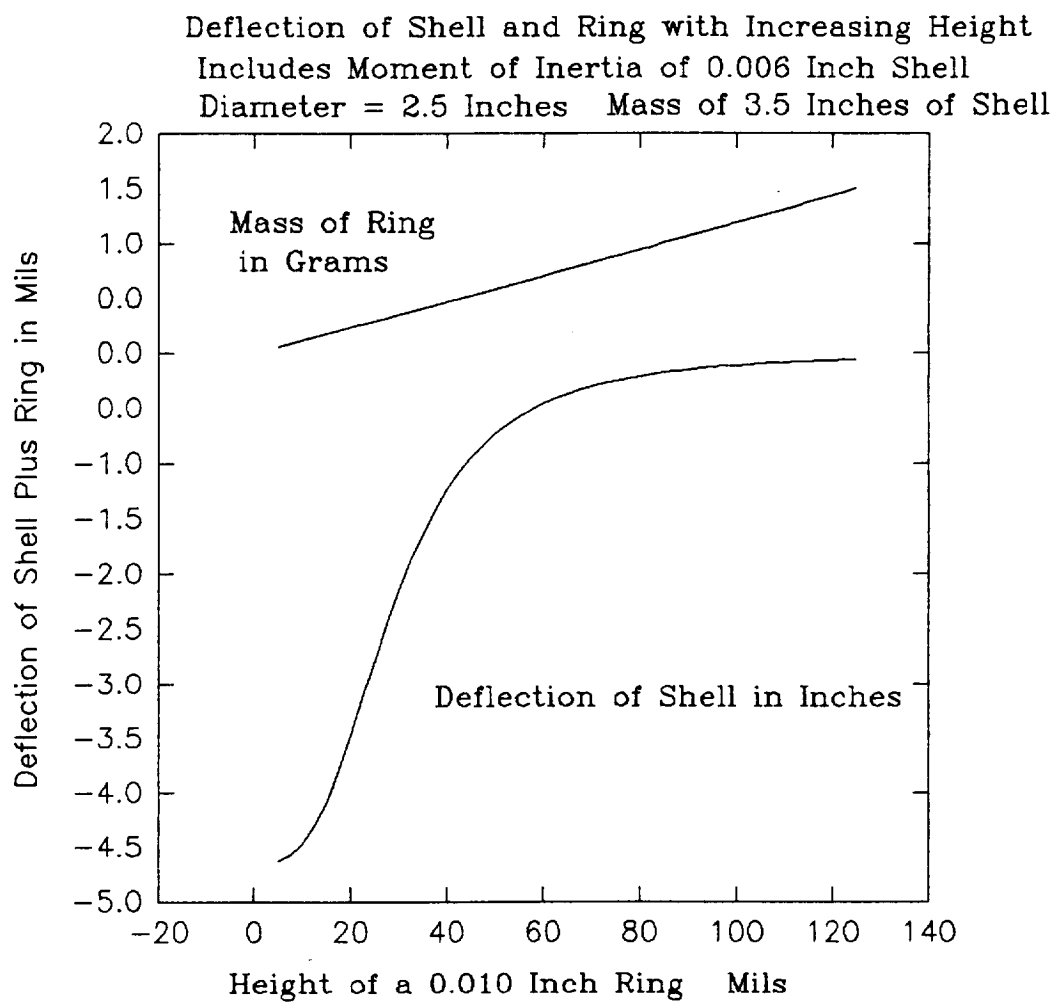


FIGURE 2

Vertical Deflection of Cylindrical Shell By
Self Weight as Thickness and Mass Increase

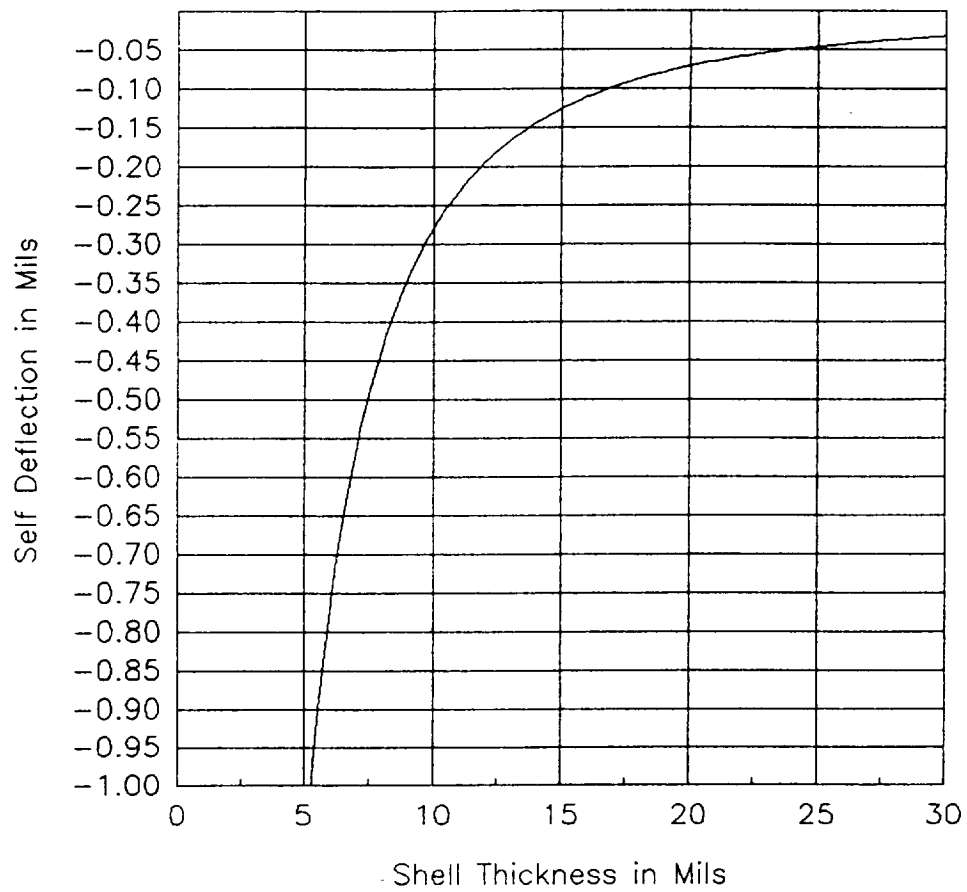


FIGURE 3

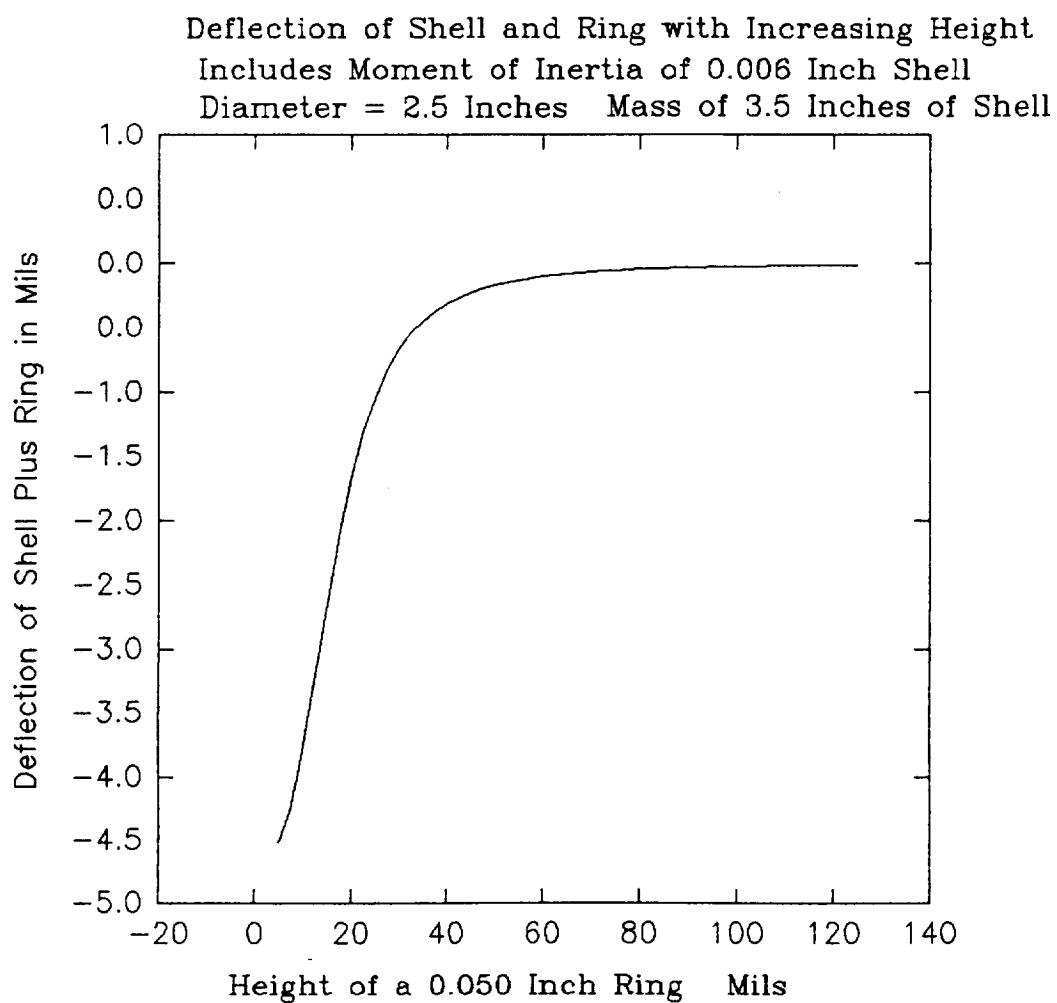


FIGURE 4

XRCF Shell Deflection Times 500 in Centimeters
1.0 G Load and 0.0154 Centimeter Thick (0.006 In)

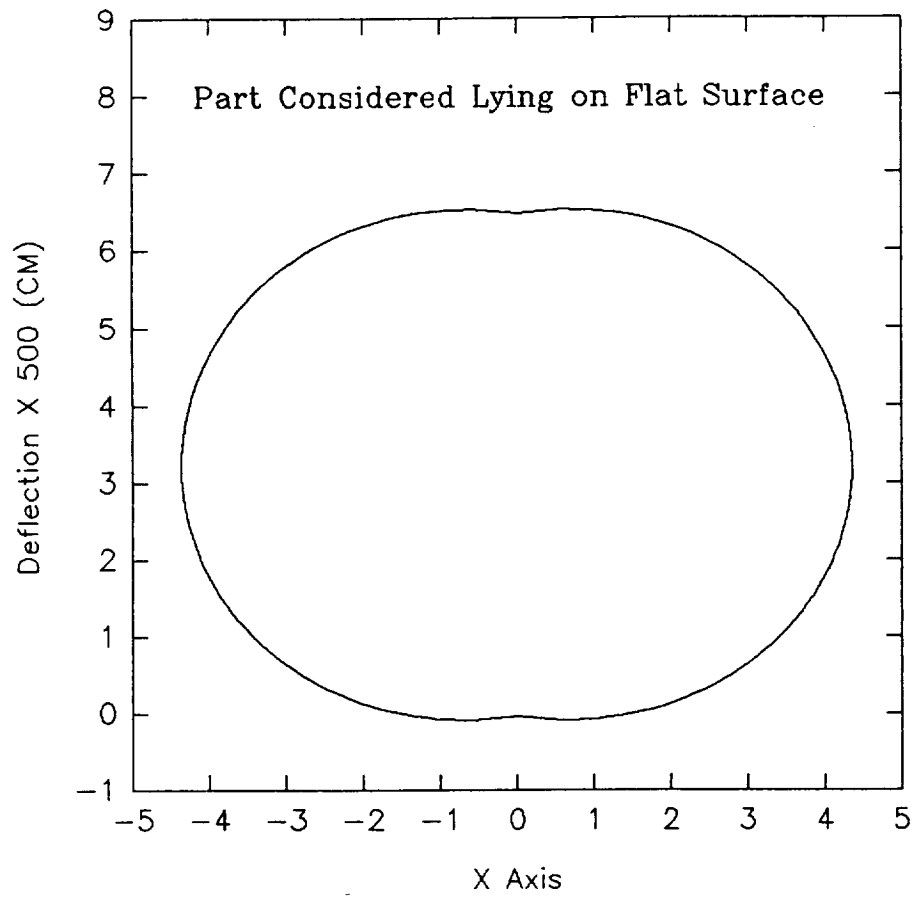


FIGURE 5

Relative Shell & Cap Weight

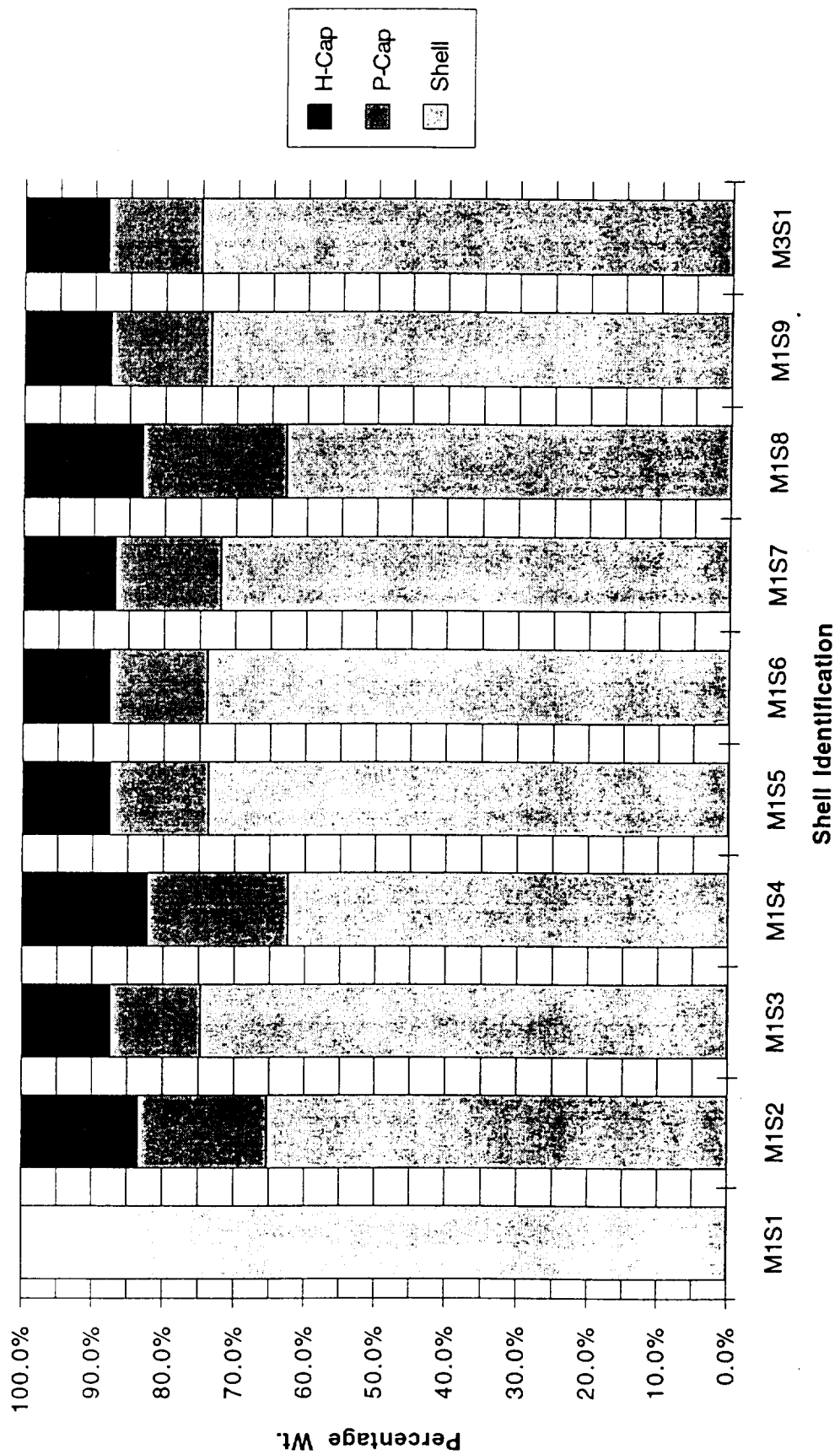


figure 6

Shear Stress at Interface of XRCF Shell to Mandrel
Zero Stress Obtained at 112 Deg. Fahrenheit

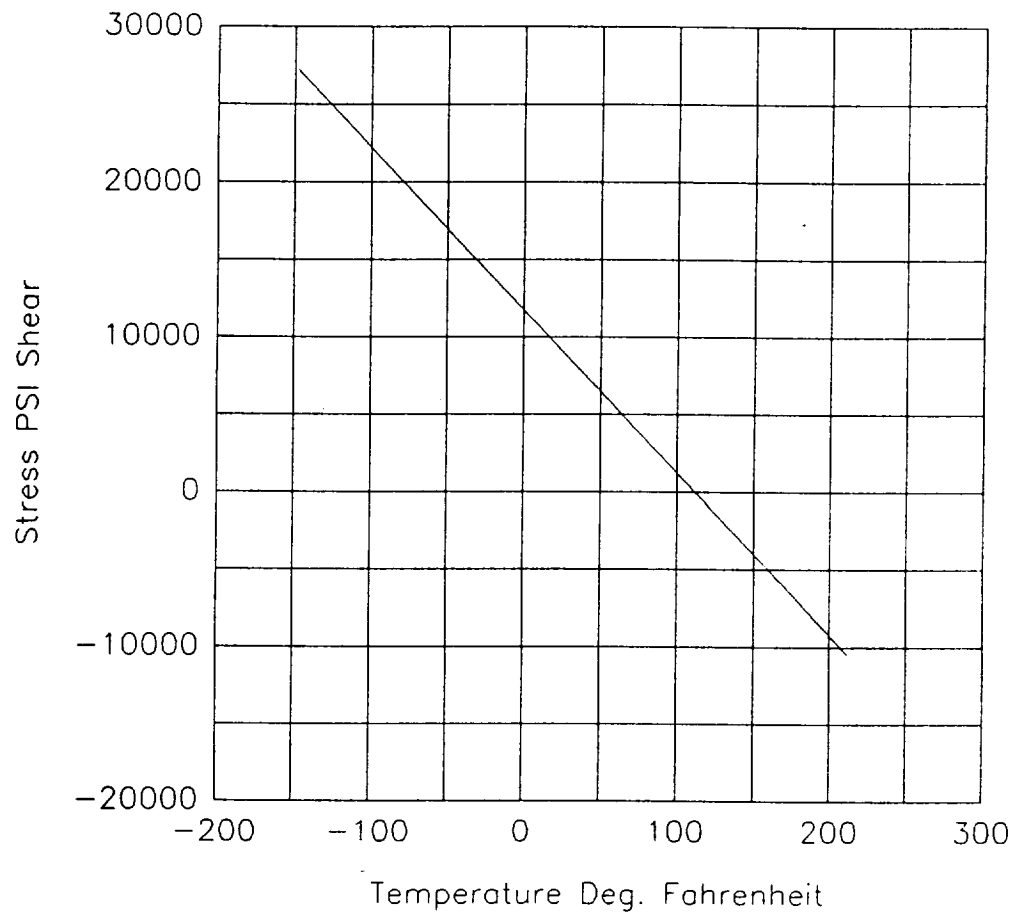


FIGURE 7

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Pi = 3.14159265
Rho = 8.90
Radius = 5.0    'I.D.
E = 1.933E9
Thick = 0.0154
Rave = Radius+Thick/2
R = Radius
Length = 1.0
Col(1) = Data(90.0,180.0,1.0)
Theta = Col(1)
Mass = 2*Pi*Rave*Thick*Rho*Length
Inertia = (Length*Thick^3)/12
X = R*Sin(2*Theta)
Y = R*Cos(2*Theta)+R
Col(2) = Y
Col(3) = X
Col(9) = -X
K1 = 0.4674*Mass/(E*Inertia*360)
K2 = -0.4292*Mass/(E*Inertia*360)
Ydefl = K1*(R-X)
Xdefl = K2*(R-Y)
Rdefl = Sqrt((X+Xdefl)^2 + (Y+Ydefl)^2)
Col(4) = Ydefl
Col(5) = Xdefl
Col(6) = Y-Sum(Ydefl)*100
Col(7) = X+Sum(Xdefl)*100
Col(8) = -Col(7)
Col(10) = Rdefl

```

FIGURE 8

APPENDIX III - Controlled Low Stress Nickel Plating Process

Discussion:

The fabrication depends on the use of very high quality small Wolter x-ray mirrors. One of the test mirrors is an electroformed nickel shell with a reflective gold inner surface. The mirror is made by the same replication processes described previously in the AXAF-S and the SXI programs using a diamond machined aluminum mandrel with 11% by weight phosphorous-nickel coating machined and polished to about 6-8 angstroms rms. The difficulty in preparing a suitable mandrel for the replication mandates that the plating be performed with extreme caution to avoid any possibility of damage to the surface.

The gold will be plated onto the nickel surface of the mandrel by electron beam evaporation with no imposed chemical passivation thereby eliminating additional risk of degrading the surface with any chemical processes for the final pieces.

The plating process for the low stress nickel is a slightly modified Barrett nickel sulfamate process from Allied Kelite. The bath has a very small amount of a stress reducing compound which is deliberately under diffusion control. By maintaining the operating conditions such that the decrease of current will allow a higher percentage of the sulfur from the additive to be incorporated as nickel sulfide within the grain boundaries, it is possible to produce a more compressive stress. By increasing the current the stress becomes more tensile due to the diffusion limited additive condition. A monitor is used to measure the real-time stress as a function of current density and time. By using computer feedback control, the stress is maintained at zero in real-time. The stress is controlled at zero for the plating temperature. It has been considered that the stress should be offset to accommodate the shrinkage back to room temperature. However this will cause the part to have some intrinsic stress or stored energy as such. This in turn is probably not desirable. The hoop stress has been estimated at lower temperatures assuming a rigid shell and zero stress at the plating temperature. The stress is possibly high enough at the interface upon just cooling from room temperature to allow separation of the gold from the electroless nickel if the adherence of the gold is low. This in turn could draw impurities under the shell before it is removed. If the shell is much thinner then the problem is reduced. Present results of testing indicate that a very clean separation is possible using gold which has been electron beam evaporated onto a very clean nickel surface cleaned in hot citric acid to remove excess oxide. This provides sufficient adhesion to avoid room temperature separation of the shell but readily permits further cooling with liquid nitrogen to allow for separation and removal.

The plating program is written in Pascal and has had all the required modules from the control card manufacturer compiled to form a single program to operate the power supply and track the major variables. The program has been modified recently to permit shutting off the power at a predetermined number of coulombs of power supply input to the plating. This permits the total nickel plated to be controlled very closely. Care must be taken to allow a small bias to remain to prevent corrosion of the shell after shutting off the power supply.

Recent experiments have indicated that the assembly of the mandrel using copper and gold plated stainless steel spring washers for contact is critical. Without the copper plating the springs would heat and drop a significant portion of the applied potential to the center or master portion of the mandrel assembly. This in turn caused the ends to overplate with nickel because of the larger portion of the current drawn. The implementation of the copper plated springs with a gold corrosion protection layer was considered adequate until additional experiments showed that the spring contact was not always intimate.

The accumulation of solution leaking past imperfect seals or the accumulation of an oxide layer on the aluminum pieces may cause the electrical connection to diminish. It is very important to assure integrity of the seals and preclean all contact points.

Nickel Plating Process:

The chemistry of the plating solution must be monitored prior to the processing of critical hardware. Important are the pH, surface tension of the liquid and the stress profile. Also the filters and the operating equipment must be checked for faults. The following conditions are prevalent:

Constituent	Condition	Tolerance	Units
Nickel Sulfamate	110	+/- 10	Grams/Liter
Boric Acid	Saturation @	105	Degrees F.
Nickel Bromide "B"	2% Vol	+/- 0.5%	
1,3,6 Naphthalene Trisulfonic Acid NTSA	Stress @ Zero 0.25	(as Required)	12-20 mA/Cm ² Grams/Liter
Sodium Laurel Sulfate "SNAP"	0.25	+/- 0.5	Grams/Liter
30-45 Dyne Cm Surface Tension by four inch ring 15 sec.			
pH	4.0	+/- 0.2	- log H _{activity}
Temperature	110	+/- 0.5	Degrees F.
Filters	2 ea. 16" 10 micron 1 ea. 16" 1 micron		Bx- 3000 Bx- 1200

Leach all filters in a solution of 0.25 grams/Liter sodium laurel sulfate (SNAP) and 2 - 4 grams/Liter sulfamic acid (SNAC). Rinse thoroughly prior to use.

Use only deionized (D.I.) water for rinse, make-up and additions.

Stress profile is to be determined at 5, 10, 15, 20, 25 mA/Cm² per Stress Monitor Operating Instructions. Zero stress should occur between 12 and 20 milliamps per square centimeter. If the current density (C.D.) is lower than 12 mA/Cm² at zero stress add NTSA in 0.05 Gm/L increments until corrected. Retest after 4 hours. Do not add more than 0.25 Gm/L without approval of process engineer. If the C.D. is higher than 20 mA/Cm² at zero stress, notify process engineer for instructions.

A test of the stress should be conducted after any significant process change or at least once a month.

The computer program "XRCF.exe" is to be called from XRCF directory by autoexecute batch file and loaded from compiled version only.

Call "XRCFset.dat" for 30 mil deposit or "XRCFset1.dat" for 6 mil deposit data file from XRCF directory only.

Set printer and data save (name file to save) options prior to starting the control.

Mandrel preparation:

Clean the rotator and all work areas prior to proceeding.

The end pieces have gone through an evolution to attempt to satisfy rough machining, diamond machining of the aluminum, electroless nickel (EN) plating, diamond turning of the EN, gold plating in either the vacuum electron beam, or the electrolytic plating process and eventually the electrolytic nickel electroforming. If one or both of the end pieces are nickel plated but not gold plated, the nickel plated piece must be passivated by a 5 minute immersion in 5 Gm/L potassium dichromate then rinsed and dried prior to assembly.

The mandrel assembly is to include Harkness MP 600 Urethane or DuPont Kalrez Teflon gaskets and a stainless steel center bolt, washer and nut. Assemble the mandrel after careful inspection of the aluminum mandrel surfaces and gold plus copper plated wave washers to avoid poor electrical contact.

Assemble mandrel with additional person available to assist.

Do not insert center bolt past the center of the threads on the large end to avoid interference with the rotator support shaft.

Place the part on the rotator using the urethane seal.

Rinse the gold plated mandrel with deionized water only.

Plating Procedure for XRCF:

Verify preparation of computer as described above.

Check solution temperature to assure that it is 110 degrees Fahrenheit. If change is required wait until it is reached prior to proceeding.

Verify that the line power to the power supply is OFF.

Measure gage body and membrane total thickness prior to starting.

Place stress monitor in solution and set controller to zero after at least 15 minutes immersion and prior to any plating tests or plating of parts.

Turn KEPCO power supply computer mode switch to manual and set both voltage and current to zero.

Place part in bath taking care to align the rotator on the predetermined marks and clamp with "C" clamps. Connect cathode lead to rotator. Turn rotator on to 50% after plugging in.

Wait one minute prior to applying plating current.

Verify that stress monitor is set to zero at this time and that the Voltage and Current controls are also at zero.

Turn power supply On - Off switch to On.

Verify Auto - Manual switch is set to Manual and slowly apply power.

Watch very carefully to set voltage to less than full scale but above value which will allow five volt compliance minimum. The ten turn control is 6.0 volts full scale. Eight to eight and one half turns are required. The power supply crowbar will trip if voltage exceeds six volts.

Adjust current to 8 amps on panel gage. Turn computer control on with supply switch still in manual. Operate for 30 minutes by controlling current manually to provide slight tensile stress which will gradually rise from zero to about one millivolt reading.

Reset stress to zero and set manual/automatic switch to automatic. Adjust Current control upward 1/2 to 1 turn to allow additional current capability for automated control. Computer will stop automatically at predetermined plating mass (determined by time and current product summation).

Procedure for changing gages in operation:

If the desired deposit thickness is more than 0.02 inches the gage should be changed at the mid point of the plating run or at 0.02 inches whichever occurs first.

- 1) Verify second gage is stripped and clean before proceeding.
- 2) Place gage in solution and clamp to prevent dropping.
- 3) Allow at least 15 minutes for temperature to stabilize.
- 4) Turn power supply Current control CCW until power supply is limiting plating current.
- 5) Set power supply computer mode switch to manual.
- 6) Swap gages and immediately reset stress "Zero" dial to zero.
- 7) Return power supply computer mode switch to automatic.
- 8) Return Current control to previous setting.

APPENDIX IV - Electrochemical Study and Current Density Results to Date

Figure 1 XRCF Calibration Mirror Plating Setup

Figure 2 10 Inch Prototype Mandrel Plating Setup

Figures 3 through 8 show improvement in distribution of deposit with shielding.

Figures 9 and 10 show the polarization characteristics of the plating solution.

XRCF X-RAY MIRROR PLATING

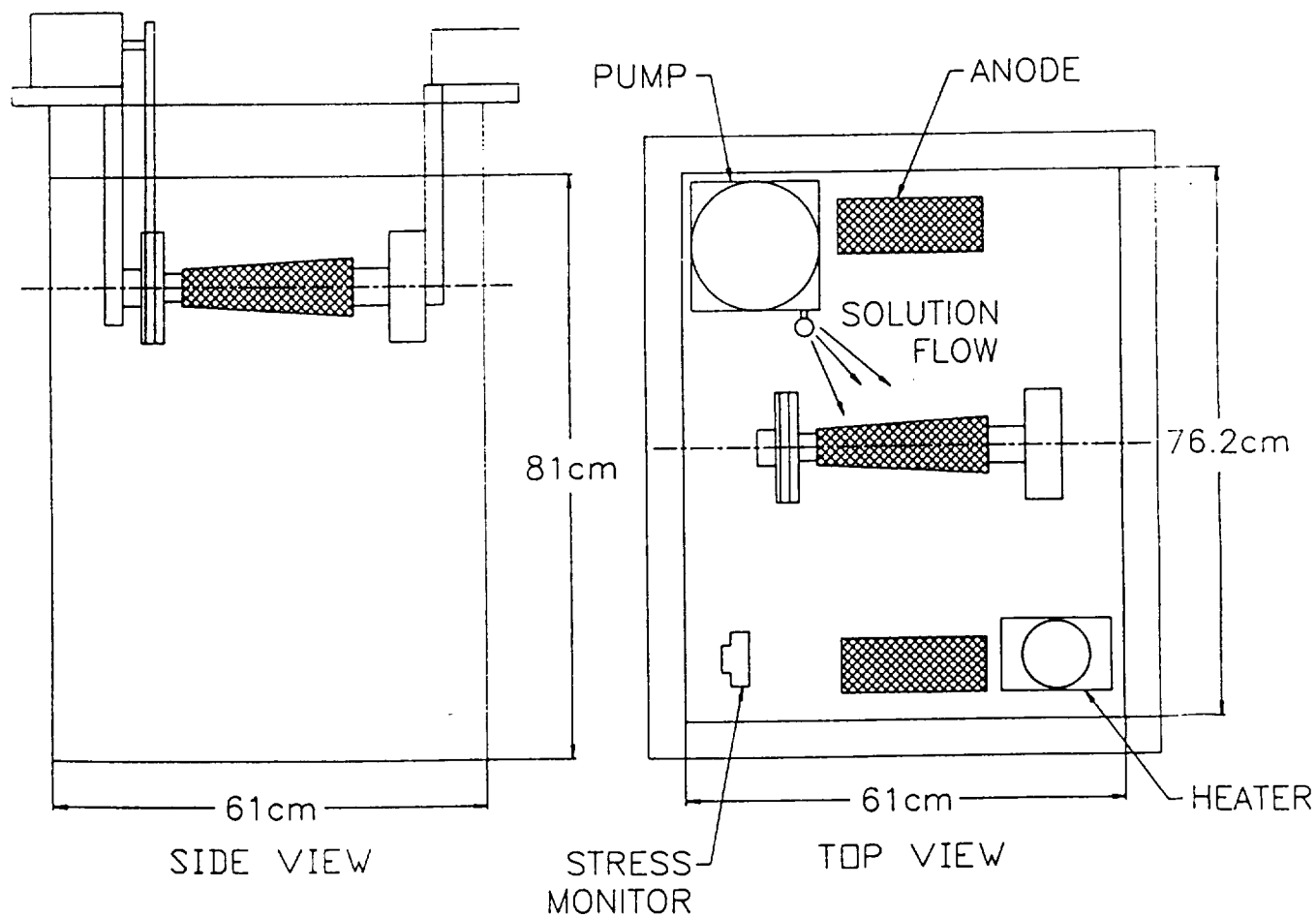
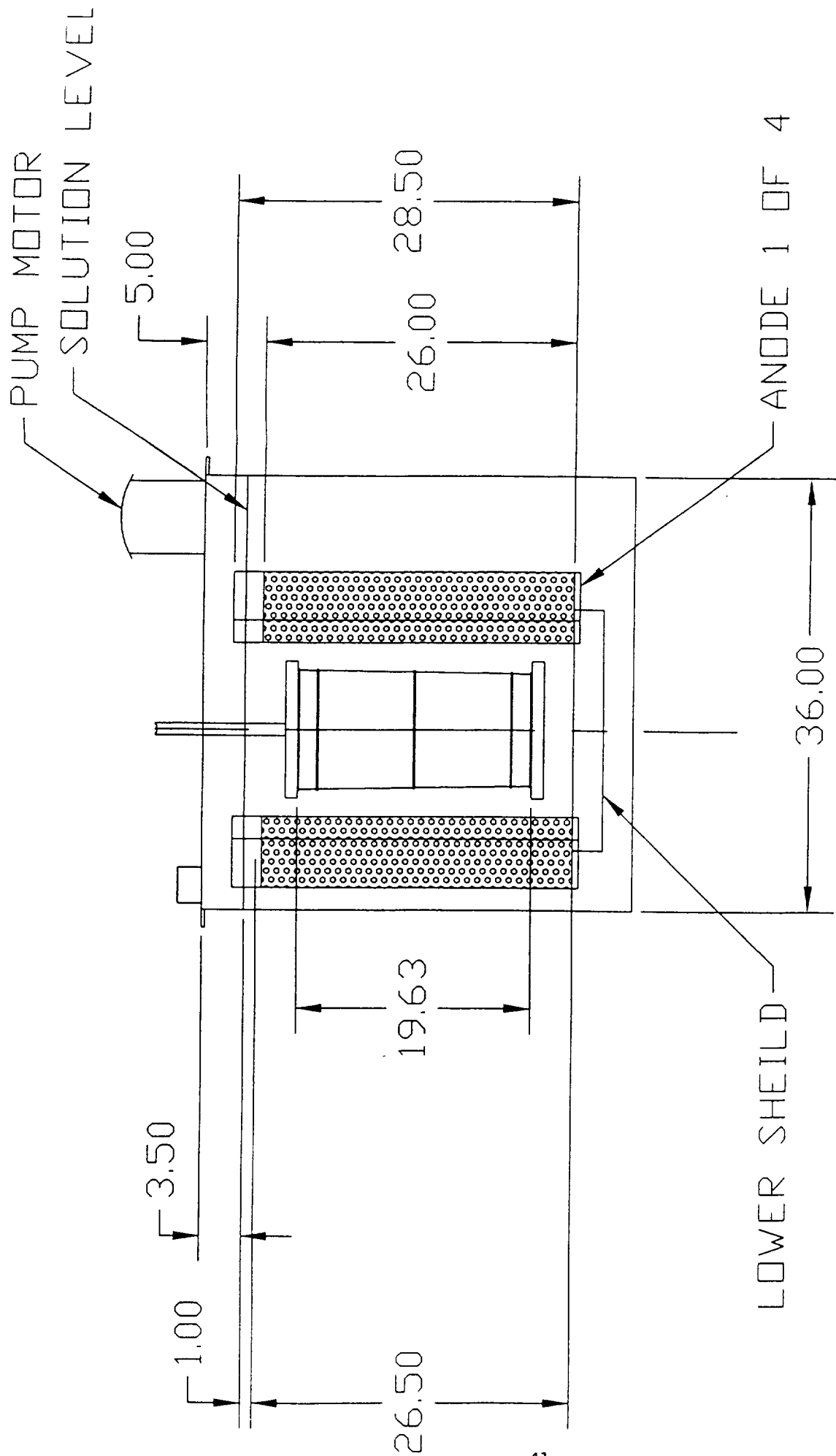


FIGURE 1



Dimensions in inches

FIGURE 2

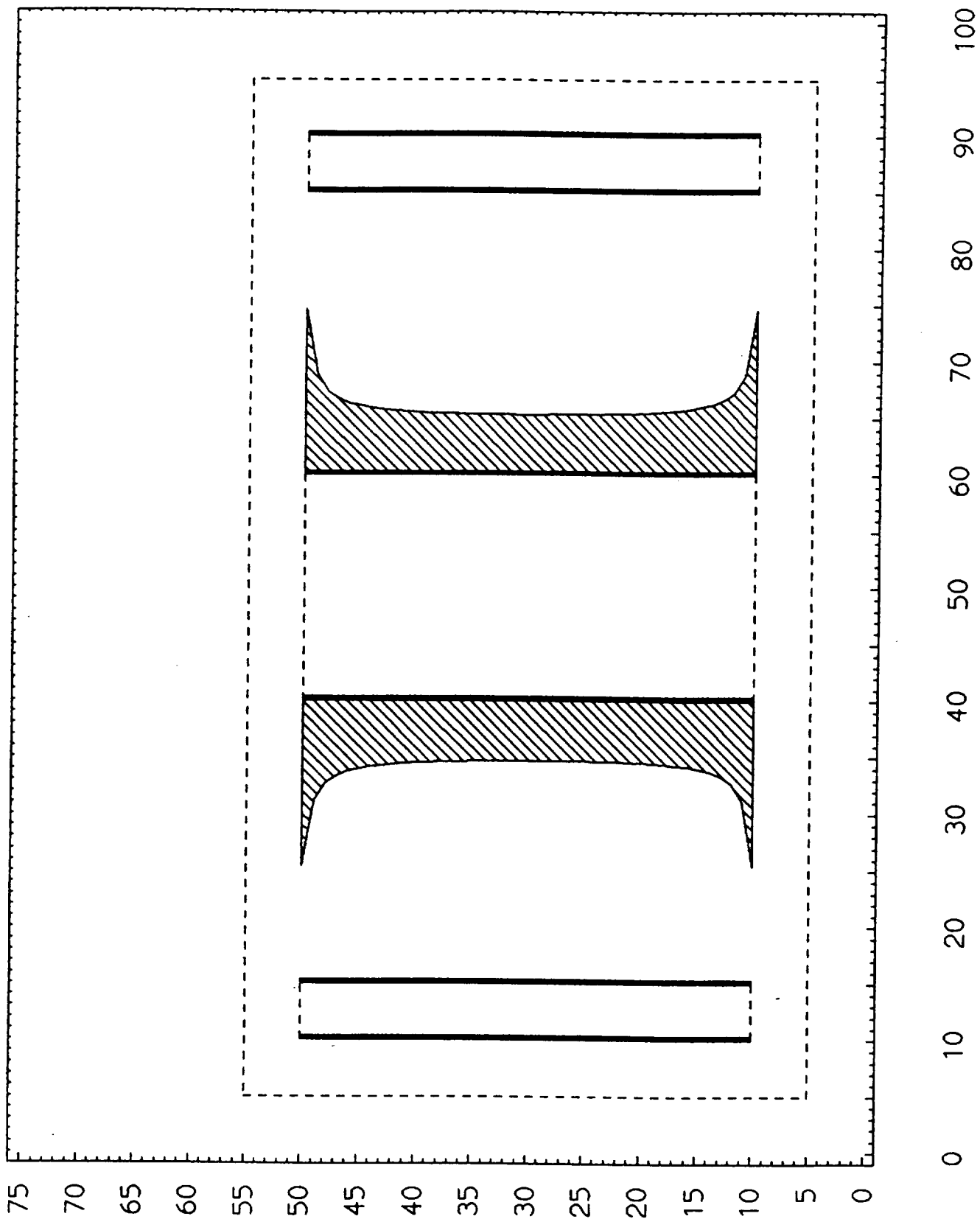


FIGURE 3

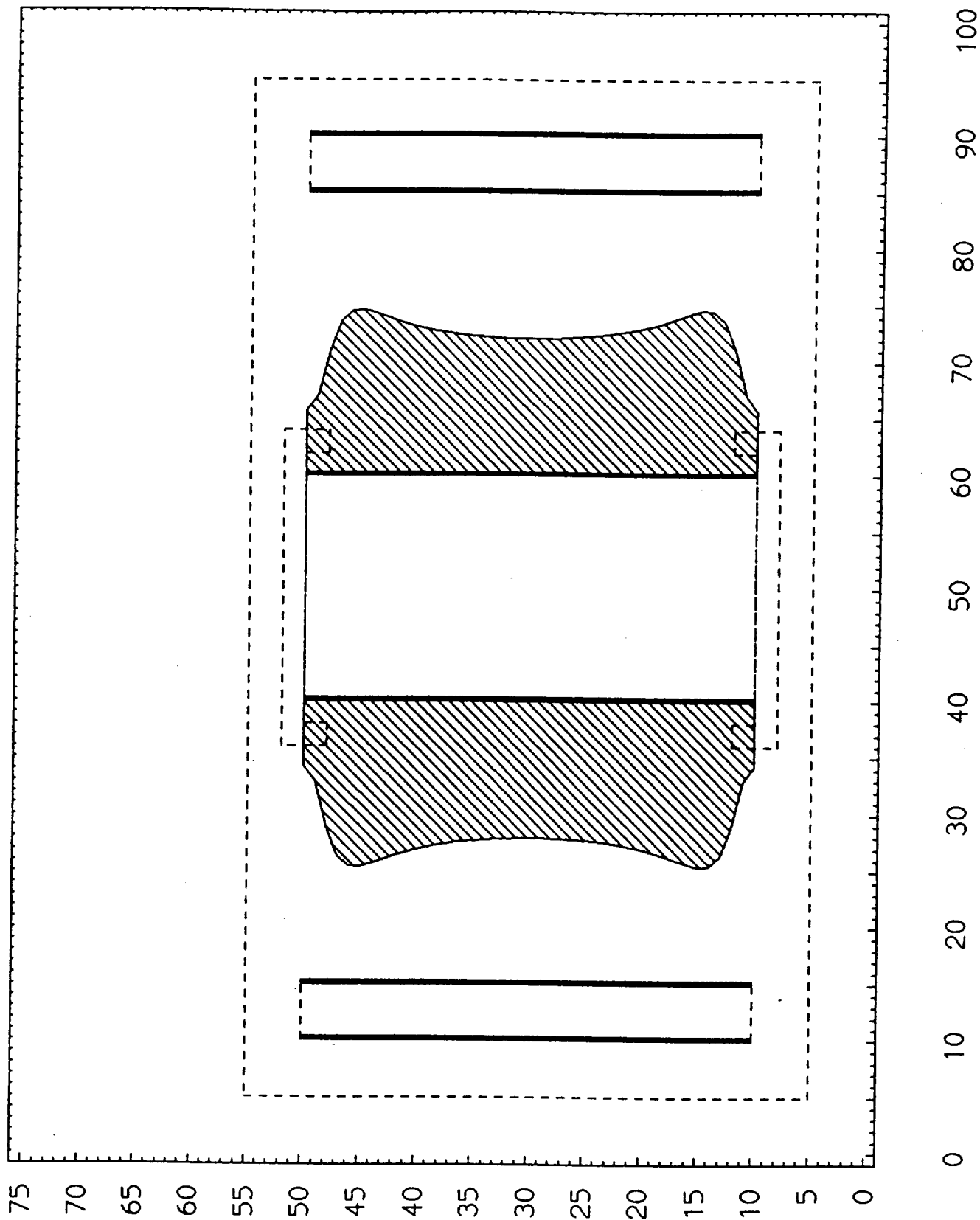


FIGURE 4

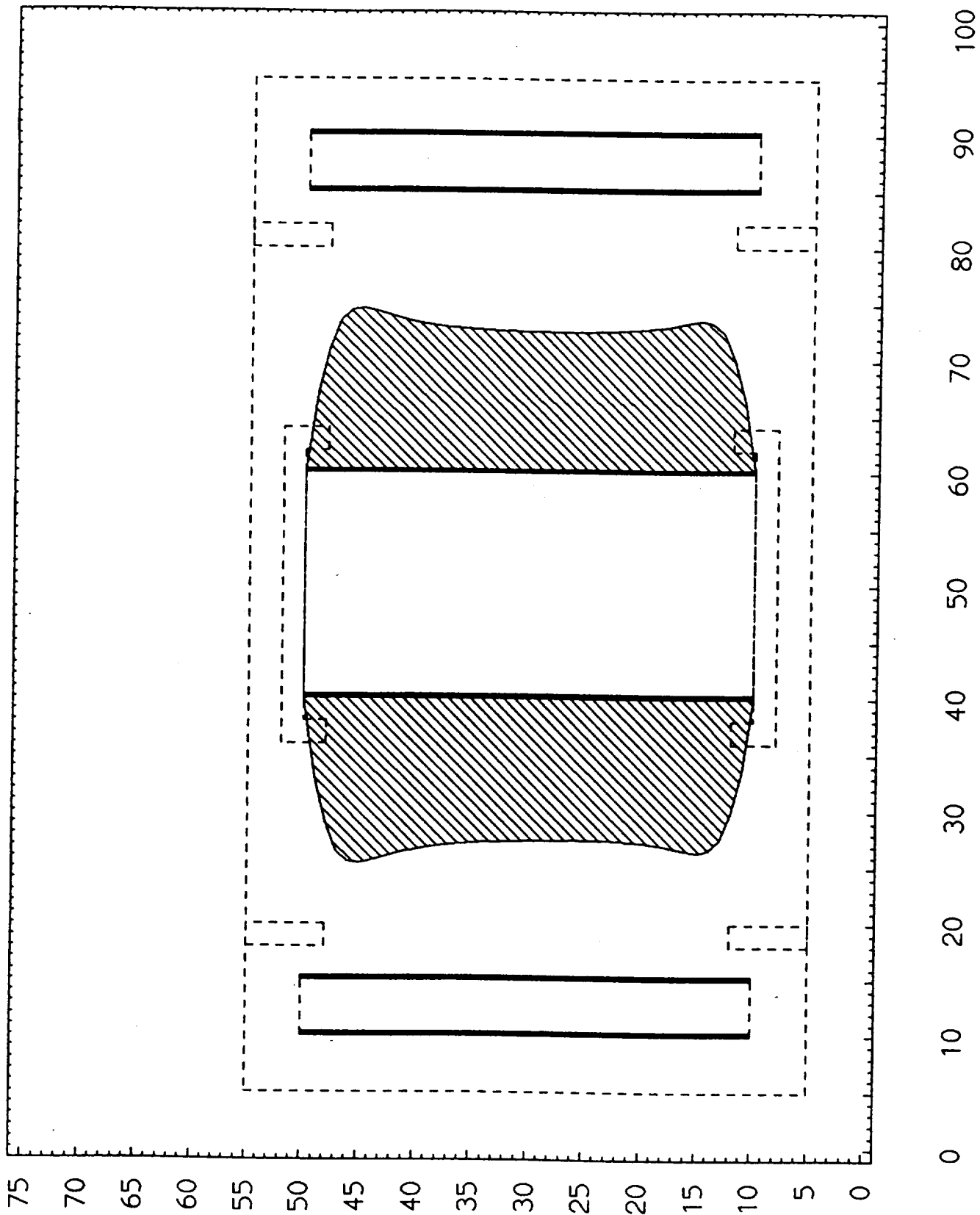


FIGURE 5

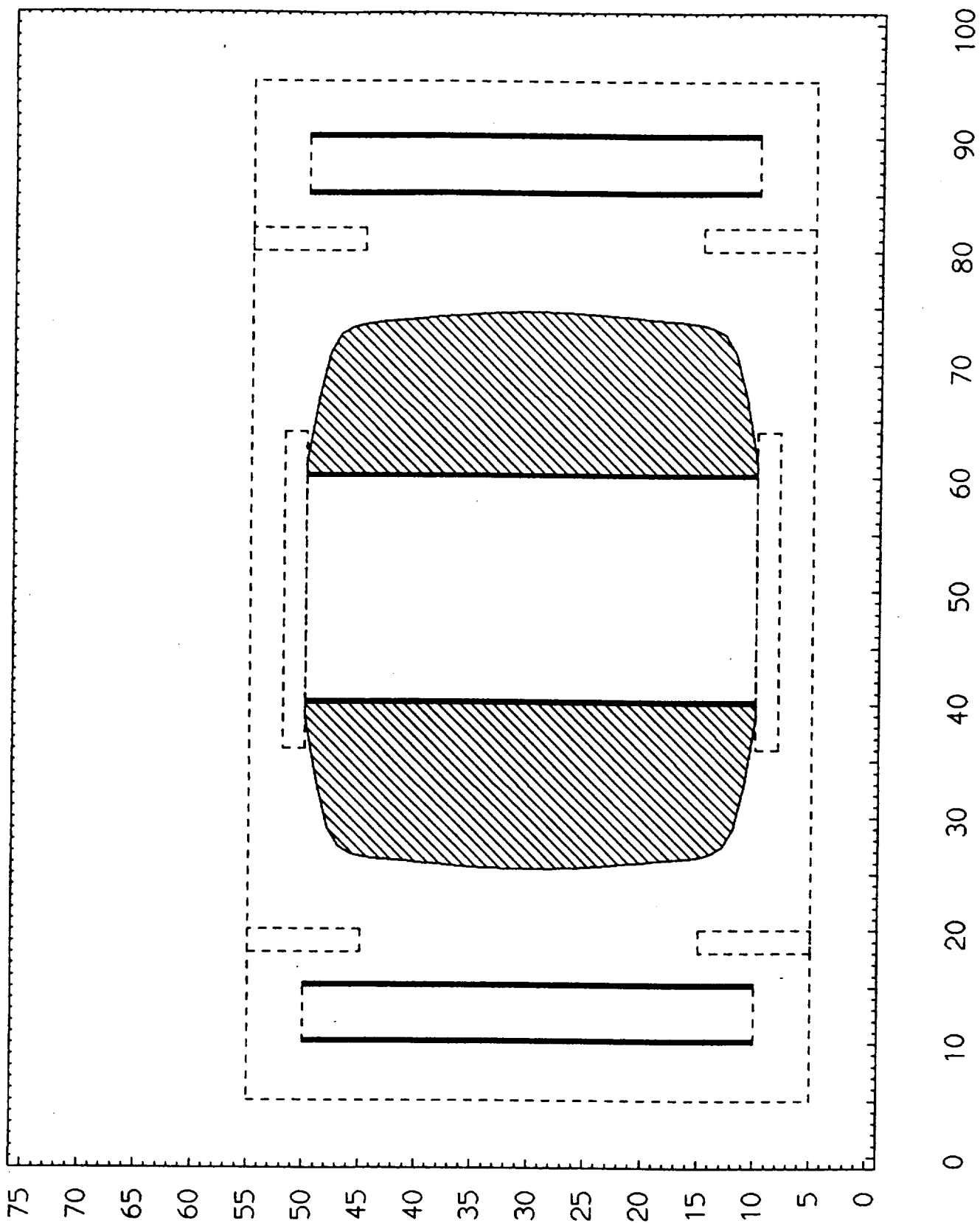


FIGURE 6

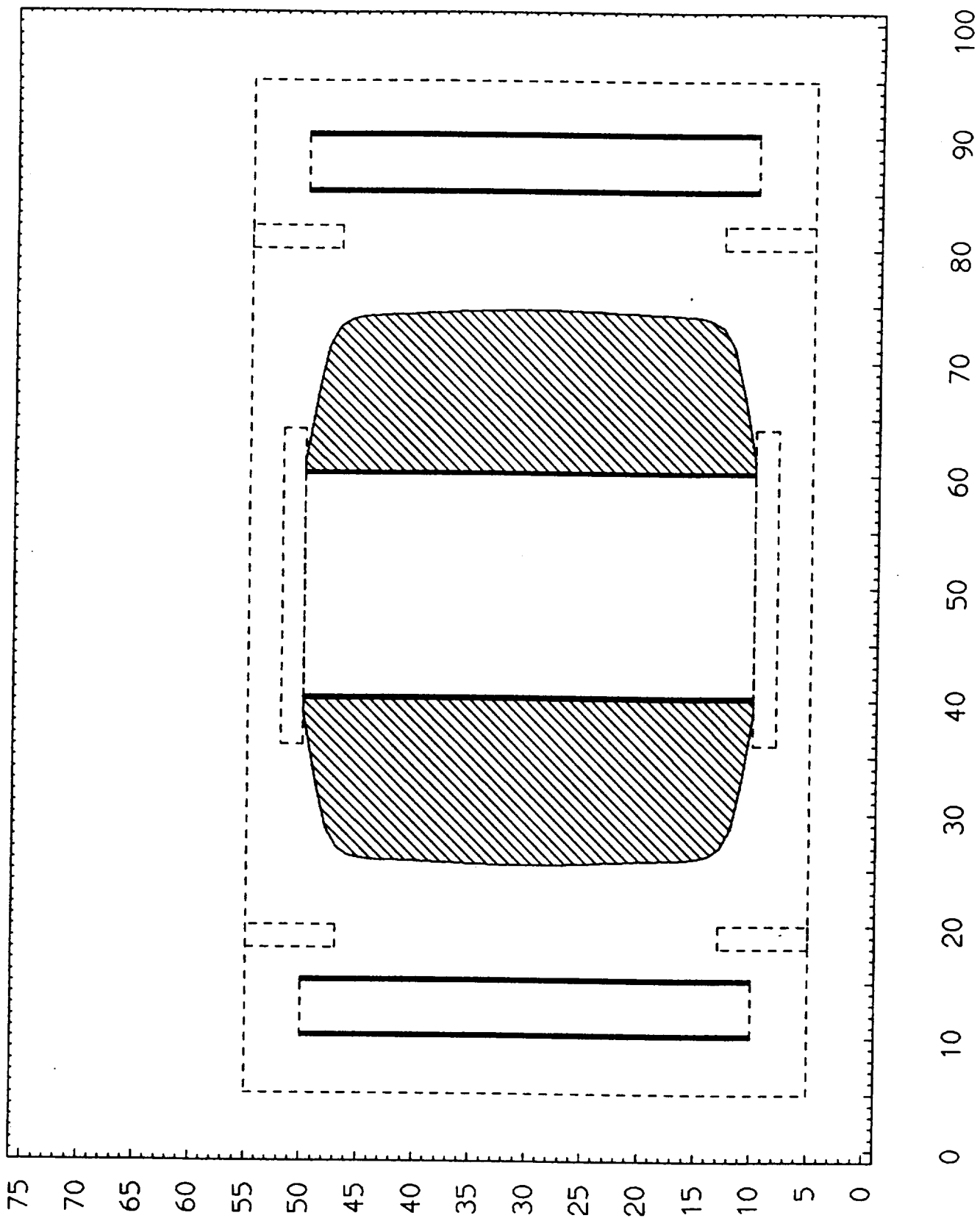


FIGURE 7

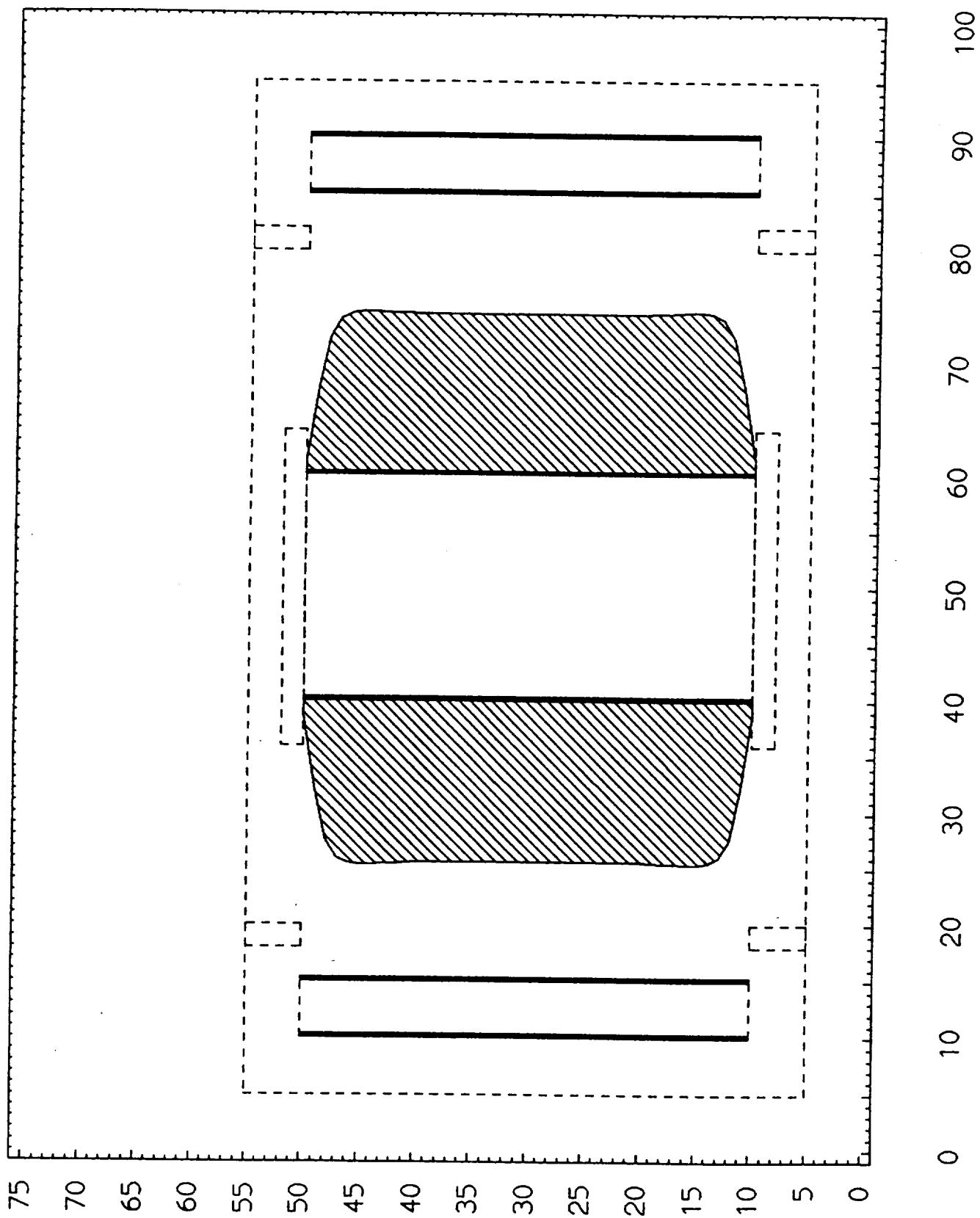


FIGURE 8

Nickel Sulfamate Polarization
43°C Stirred

$$B_a = 0.2199$$

$$B_c = 0.1097$$

$$E_{\text{corr}} = 0.6011$$

$$I_{\text{corr}} = 11.52 \mu\text{Acm}^2$$

Model 352 Corrosion Analysis Software, v. 1.00		LP LINEAR POLARIZATION		FILE STATUS: NORMAL	
DATE RUN: 12-24-96 TIME RUN: 04:16:43					
CP -1.100 vs. R	CT 240	IP -1.000 vs. R	ID PASS	FP -0.250 vs. R	SI 5.000E-04
SR 1.000E-03	ST 5.000E-01	CR AUTO	NP 1501	IR NONE	FL NONE
RT HIGH STABILITY	REF 0.24150 SCE	WRK SOLID	AR 1.000E+00	LS NO	EN 5.870E+01
DEN 0.000E+00	OC -0.684				

— sh3002.dat

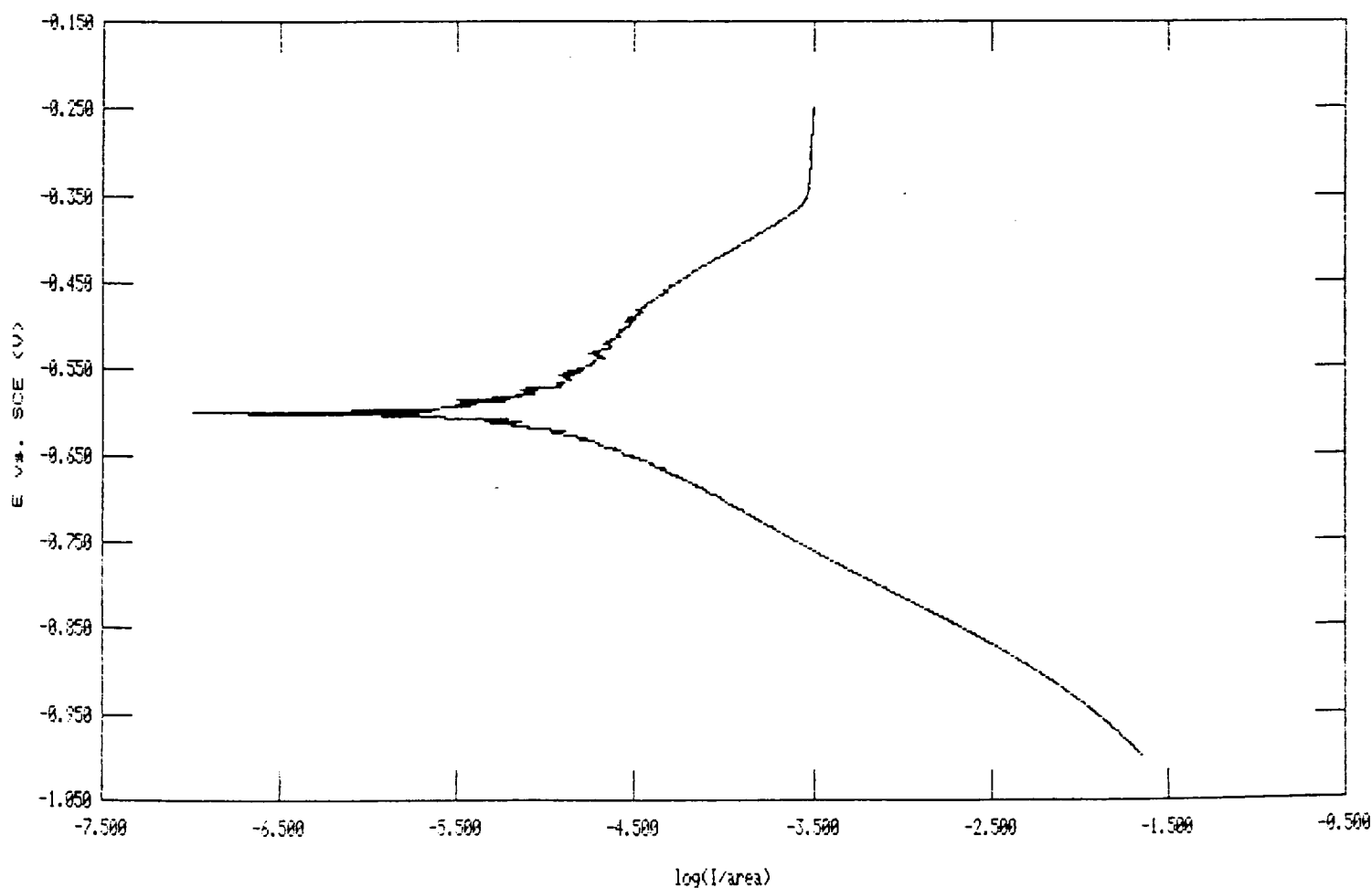


FIGURE 9

Nickel Sulfamate Polarization
63.5°C Stirred

$$\begin{matrix} B_a = 0.1729 \\ B_c = 0.1046 \end{matrix} \begin{matrix} \diagup \\ \diagdown \end{matrix} \begin{matrix} \\ \end{matrix} \text{V / decade}$$

$$E_{\text{corr}} = 0.5730 \text{ V}$$

$$I_{\text{corr}} = 32.16 \mu\text{A}/\text{cm}^2$$

Model 352 Corrosion Analysis Software, v. 1.00		LP LINEAR POLARIZATION		FILE STATUS: NORMAL	
DATE RUN: 12-24-96 TIME RUN: 02:39:41					
CP -1.100 vs. R	CT 240	IP -1.200 vs. R	ID PASS	FP -0.250 vs. R	SI 5.000E-04
SR 1.000E-03	ST 5.000E-01	CR AUTO	NP 1501	IR NONE	FL NONE
RT HIGH STABILITY	REF 0.24150 SCE	WRK SOLID	AR 1.000E+00	LS NO	EW 5.870E+01
GEN 8.000E+00	QC -0.631				

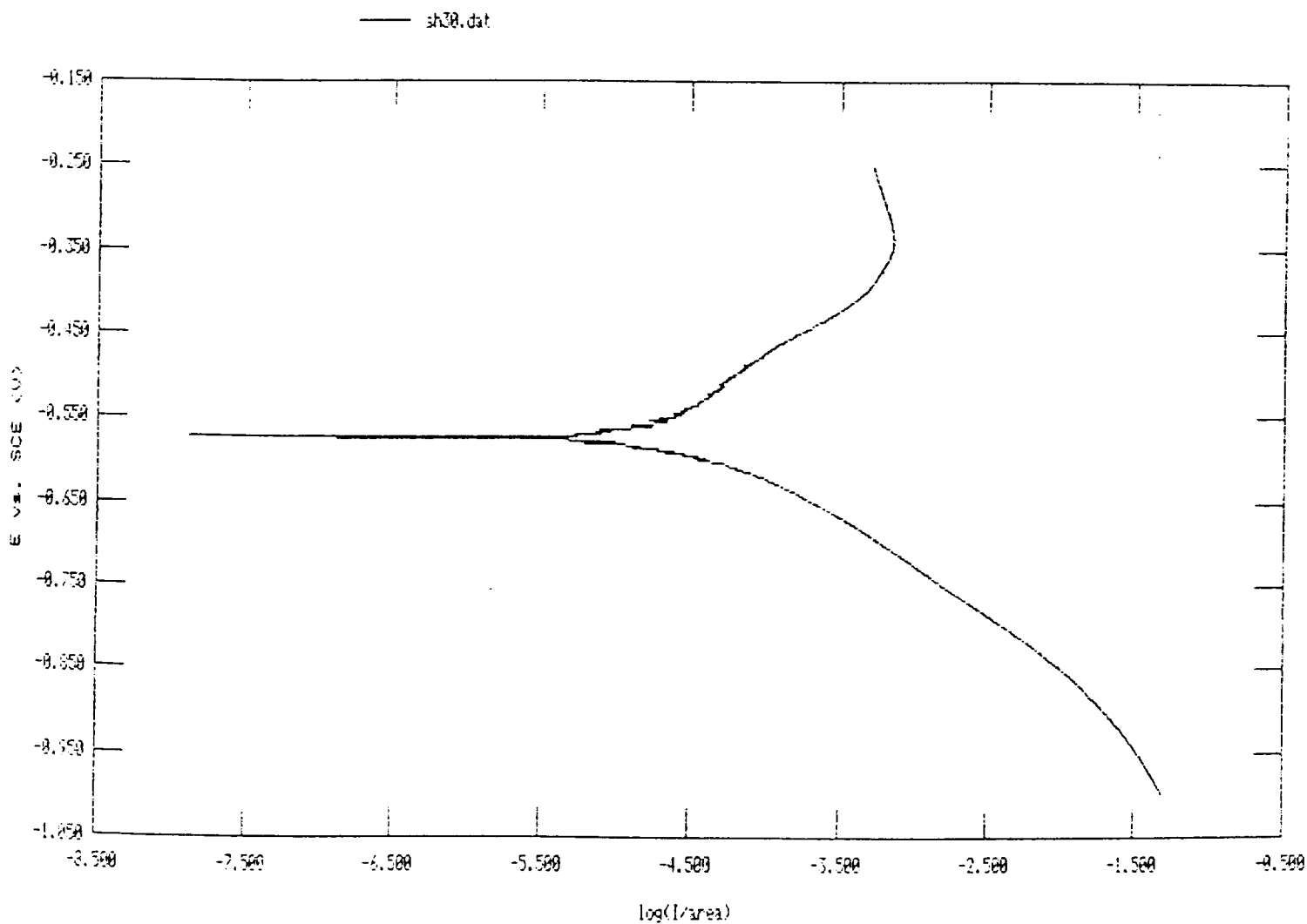


FIGURE 10

Nickel Sulfamate Solution (Standard Barrett to 110 gm/L Nickel)

C.D.	15/mA/cm [^]	Nominally controlled to produce zero stress in real time
Temperature	45 Deg. C.	
Conductivity	0.049 S/cm	
Rho Nickel	8.90 gm/cm ^{^3}	
At. Wt.	58.7	
Z	2	
Alpha	1.8	(Lchem)
Beta	0.2	(Lchem)
Concentration	1.87	Molar in nickel
Transport # Ni	0.35	Estimated based on data from other similar processes
Efficiency (C)	98.0% Ni + (2% Hydrogen & Other Reactions)	
Efficiency (A)	100.0% Ni	
J exchange	1.2 * e ⁻⁵	amps/cm ^{^2}
EMF	- 0.800 V	vs SCE
OCP	- 0.601 V	vs SCE
Diffusivity	1.0 * e ⁻⁵	cm ^{^2} /s
J limiting	200 mA/cm ^{^2}	
v (viscosity)	1.5 cp	
Re	2000	
Ta	5.6 * e ⁻⁶	
Tafel Slope (C)	104.6 mv/Decade	(measured)

With these values (when applicable), the Lchem program seems to give reasonable values.

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